2330

STABILITY STUDY:

4-AAP PHENOLICS IN

PRESERVED SAMPLES

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PIBS 2330

STABILITY STUDY:
4-AAP PHENOLICS IN PRESERVED SAMPLES

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ENVIRONMENT ONTARIO

SEPTEMBER 1992
for the MISA
Joint Analytical Working Group (JAWG)
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INTRODUCTION

The MISA Industrial Monitoring Regulation 695/88 as amended to Ontario Regulation 533/89 under the Environmental Protection Act, Effluent Monitoring - General, requires a maximum storage time of only 4 days for ATG 14 (analytical test group), 4-AAP phenolics. This allows little time for the delivery and analysis of samples. In order to determine if there was sound basis for the extension of the storage time, seven members of the MISA Industrial Sector's Joint Analytical Working Group (JAWG) agreed to participate in a study of the stability of reactive phenolics in preserved samples over time.

The study was done in two phases. The first was undertaken by five independent laboratories and involved the storage and analysis of preserved solutions of phenol, spiked in distilled deionized water, over a period of approximately 30 days. It was agreed that if no significant change was detected participants would proceed with the second phase; the storage and analysis of preserved effluents containing significant levels of reactive phenolics, as is and spiked with pure phenol. The second phase was undertaken by six independent laboratories, representing five MISA Industrial sectors* and the MOE. One or more effluents from each sector were studied.

* All MISA Industrial sectors were invited to participate; the five represented were as follows:

Inorganic Chemical	(ICI)
Iron & Steel	(DOFASCO)
Organic Chemical	(DOW CHEMICAL CANADA INC.)
	(POLYSAR RUBBER CORP.)
Power Generation	(ONTARIO HYDRO)
Petroleum Refining	(NOVACOR)

PROCEDURE

PHASE ONE:

Phenol solutions of 10 and 40 ug/L in distilled deionized water were prepared by each participant. A minimum of ten glass or Teflon^{R} sample bottles with phenolic-free caps were filled with each of these solutions; five at each concentration were preserved with H_2SO_4 to a Ph between 1.5 and 2.0; five at each concentration were preserved with 1mL of 3N H_3PO_4 + 120g/L $CuSO_4.5H_2O$ solution, for each 250 mL of sample.

For the duration of the study, samples were stored in the dark at 4 to 10°C. A minimum of one aliquot from each bottle was analysed every two to five days over a period of approximately 30 days.

PHASE TWO:

Effluent samples were collected by participants from each industrial sector. The objective was a minimum of one representative effluent per sector, containing measurable phenolics and considered to be a complex, possibly biologically active matrix.

Each effluent was split into two portions; one was left unadulterated and one was spiked with 20 ug/L phenol. A minimum of six glass or Teflon^{R} bottles with phenolic free caps were filled with each of these solutions; three of the unadulterated effluent and three of the spiked effluent were preserved with H_2SO_4 to a Ph between 1.5 and 2.0; three of the unadulterated effluent and three of the spiked effluent were preserved with 1mL of 3N H_3PO_4 + 120g/L $CuSO_4.5H_2O$ solution, for each 250 mL of sample.

For the duration of the study, samples were stored in the dark at 4 to 10°C. A minimum of one aliquot from each bottle was analysed every two to five days over a minimum of 30 days.

Controls consisted of a blank and spiked blank (20ug/L) for each preservation technique, prepared, stored and analysed in the same manner as the effluent samples.

RESULTS AND DISCUSSION

PHASE ONE

Data means and standard deviations, for each day of analysis, by each of the five analytical laboratories participating in this phase, are shown in **Table 1** and are graphically represented in **Figure 1A** (H_2SO_4 preservative) and **Figure 1B** ($CuSO_4/H_3PO_4$ preservative) with control limits, about the expected value, of +/- 3SD, based on the mean(all data)SD at each spiking level, for each preservative.

The precision of measurements varied between laboratories, however the data clearly show that the preserved phenol solutions remained stable over the 30 days of the study. Results fell within the 3SD control limits about the expected value, over the course of the study, except where lack of control over precision was apparent and excursions outside control were seen from day one, for some laboratories.

There was statistically similar behaviour for phenol solutions preserved with H_2SO_4 (S) and those preserved with $CuSO_4/H_3PO_4$ (CP).

Precision:
At 40 ug/L the mean SD of (S) data was 0.82 ug/L and (CP) data 0.85 ug/L.
At 10 ug/L the mean SD of (S) data was 0.61 ug/L and (CP) data 0.57 ug/L.

Stability:
The combined lab means, based on a minimum of five replicates/laboratory were, for
DAY 00 : (S)40.74 ug/L (CP)40.30 ug/L (S)9.92 ug/L (CP)9.65 ug/L, and for
DAY ≥ 30: (S)40.73 ug/L (CP)39.19 ug/L (S)9.65 ug/L (CP)9.61 ug/L.
All fall within one SD of the expected values 40 ug/L and 10 ug/L.

PHASE TWO:

Data for each effluent studied, along with calculated means and SD's are shown in **Tables 2** through **13**. The corresponding **Figures 2** through **13** give a graphical representation of the mean values for the various effluents, spiked effluents and blanks along with a plot of the spike recoveries and spiked blanks. The results from both preservation techniques are included on each of the graphs.

Ontario Hydro continued their analyses of four effluents from the Nanticoke facility and two from the Darlington facility, beyond thirty days. The graphical representation of these data (Fig 8 to 13) are shown with an extended time axis of 100 or 120 days, where the others (Fig 2 to 7) are over 30 to 35 days.

Measurable levels of 4-AAP phenolics were found in five of the collected effluents. They ranged from less than 1 ug/L to 50 ug/L. For one effluent, shown in Table 2 and Figure 2, the level measured, from day 00, differed by approximately 20% between effluent preserved with H2SO4 and replicates preserved with CuSO4/H3PO4. Levels were approximately 50 ug/L and 40 ug/L respectively, however, there was no significant change in these levels over the 30 days of the study.

Spike recovery from some effluents differed from the control (spiked blank) significantly, apparently effluent dependent, but for a given effluent, spike recovery did not change significantly over the 30 day study period.

In **Figure 14A** the overall stability is shown graphically with the %CHANGE of each day's mean value from the initial mean value (n=5) plotted against time, for each preservative, up to 47 days. The trend is identified using a "best-fit" trend line (Harvard Graphics). The trends are consistent; slightly negative **slope** over 47 days, for (S) data slope= -0.08, for (CP) data slope= -0.14

The stability shown is consistent with the PHASE ONE findings in that the "best-fit" trends show values at 30 days to be well within one standard deviation* of the expected value. The %CHANGE (or %difference) from the expected at **30 days** for (S) data was -1.1% and for (CP) data was -1.4%

* overall mean SD 0.84 ug/L @ the 40ug/L level = ± 2.1 % (PHASE ONE)
  overall mean SD 0.89 ug/L @ the 20ug/L level = ± 4.5 % (PHASE TWO)
  overall mean SD 0.59 ug/L @ the 10ug/L level = ± 5.9 % (PHASE ONE)

The spiked blank (control) data again showed stability over the 30 day period. The overall mean value (n=14) for the control spiked blanks on DAY 00 (initial) was 20.54 ug/L and on DAY 30 to 47 combined (n=12), 19.94 ug/L well within one standard deviation of the expected.

The additional data, provided by Ontario Hydro extending their analyses beyond 30 days, clearly shows a significant drop in phenolics measured by the 4-AAP test...but this occurs well beyond the 30 day point in all effluents studied. This holds true for those samples preserved with H2SO4 and replicates preserved with CuSO4/H3PO4. The **Figure 14B** shows the graph of % change of spiked effluents (means) over the extended time period.

RECOMMENDATIONS

1. The storage time for MISA ATG 14 (analytical test group), 4-AAP phenolics should be extended to **30 days** for samples preserved with either H_2SO_4 or $CuSO_4/H_3PO_4$, and stored at 4°C. This recommendation is not without precedent. The AWWA.APHA.WPCF Standard Methods of Analysis for Water and Wastewater require that preserved (H_2SO_4) and stored (4°C or less) samples be analysed within 28 days of collection. The EPA method 420.2 (Colourimetric automated 4-AAP with distillation) Storet# 32730 requires analysis within XX days.

2. Analysts must ensure effluents are screened and treated to eliminate major interferences to the 4-AAP test, such as oxidizing agents and sulphur compounds, as soon as possible after collection. Historic characterization of these effluents would facilitate prompt treatment.

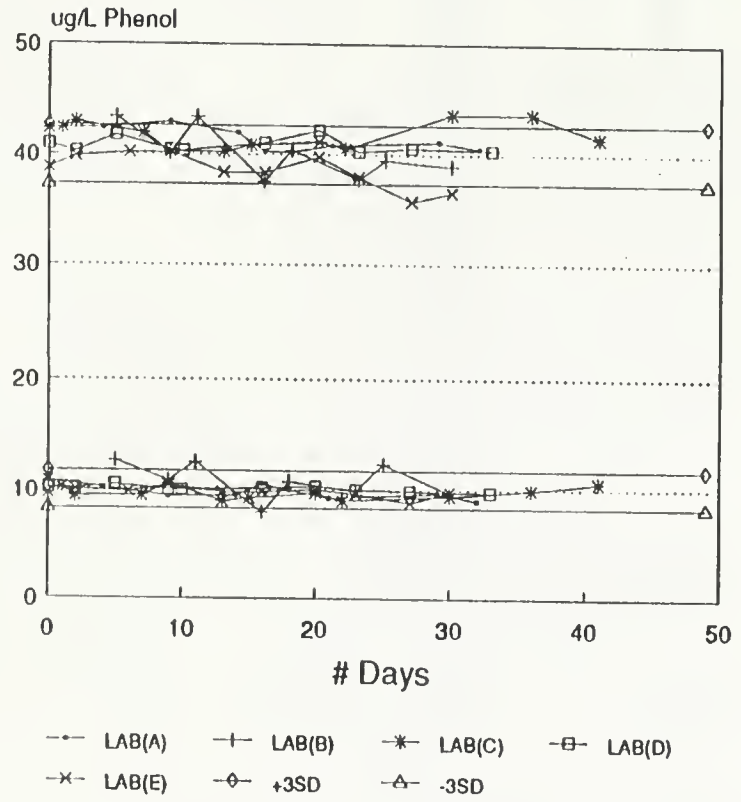
3. Analytical methodologies and instrument conditions for the 4-AAP test should be scrutinized, as study data showed significant differences for within run precision between laboratories. (See Environment Ontario .. INTERLABORATORY STUDY 92-5)

TABLE 1 PHENOL SPIKES IN DDW/ PRESERVED

PARTICIPANT		#DAYS										
LAB A (DOF)		0			4	9	14	16	18	21	29	32	
LAB B (DOW)					5	9	11	16	18	23	25	30	
LAB C (HYD)		0	1	2	7	9	13	15	20	22	30	36	41
LAB D (MOE)		0		2	5	9	10	16	20	23	27	33	
LAB E (POL)		0		2	6	9	13	16	20	23	27	30	
LAB F (NOV)		...DID NOT PARTICIPATE IN PHASE ONE.											
DDW/CuSO4 H3PO4 + 40 ug/L	(A) MEAN	41.59			41.29	41.00	39.24	40.14	39.82	40.53	41.24	38.16	
	SD	0.90			0.55	0.85	0.34	0.31	0.16	0.68	0.35	0.80	
	(B) MEAN				43.40	42.80	43.60	37.20	41.80	39.60	40.60	39.00	
	SD				1.36	1.59	2.33	2.02	0.92	0.86	1.63	1.82	
	(C) MEAN	40.43	41.33	39.67	39.40	40.49	40.34	39.45	38.98	40.68	38.70	41.51	39.78
	SD	0.54	0.08	0.64	0.73	0.40	0.35	0.85	0.91	2.08	0.86	1.11	0.91
	(D) MEAN	40.40		39.84	41.48	40.18	39.84	40.70	41.48	39.44	40.00	40.00	
	SD	0.14		0.33	0.36	0.25	0.30	0.36	0.30	0.26	0.14	0.50	
	(E) MEAN	38.80		39.80	40.20	40.20	38.40	38.40	39.80	38.00	35.80	36.60	
	SD	0.45		1.10	0.84	3.00	0.55	0.55	0.45	0.71	0.45	0.89	
DDW/CuSO4 H3PO4 + 10 ug/L	(A) MEAN	9.53			10.03	11.07	9.74	10.37	9.57	10.90	10.78	9.22	
	SD	0.48			0.54	0.33	0.70	0.22	0.59	0.78	0.25	0.84	
	(B) MEAN				12.90	11.30	13.30	7.95	11.40	10.00	11.50	9.23	
	SD				1.07	0.82	1.78	1.35	0.68	2.23	0.96	1.35	
	(C) MEAN	9.93	9.80	10.52	10.18	9.93	9.51	9.69	10.59	10.20	10.48	9.73	10.07
	SD	0.43	0.33	0.06	0.80	0.25	0.31	0.20	0.67	0.38	0.33	0.25	0.35
	(D) MEAN	9.94		10.20	10.44	9.94	9.96	10.16	10.18	9.76	10.14	9.60	
	SD	0.19		0.27	0.11	0.19	0.22	0.17	0.13	0.13	0.13	0.17	
	(E) MEAN	9.20		10.00	9.80	10.60	9.60	9.60	9.80	9.40	8.80	9.00	
	SD	0.45		0.00	0.45	0.89	0.55	0.55	0.45	0.55	0.45	0.00	
DDW/H2SO4 + 40 ug/L	(A) MEAN	40.72			41.29	41.36	38.61	39.79	40.13	38.70	41.06	40.22	
	SD	1.13			1.11	1.14	0.50	0.49	0.54	0.43	0.84	0.49	
	(B) MEAN				43.40	40.10	43.40	37.60	40.40	37.80	39.50	38.96	
	SD				0.79	1.00	0.70	1.94	2.05	1.93	1.36	1.81	
	(C) MEAN	42.31	42.41	42.97	41.96	40.28	40.26	40.86	41.17	40.57	43.66	43.64	41.49
	SD	0.69	0.74	0.60	0.71	0.35	1.77	1.49	1.23	1.53	0.51	0.68	0.39
	(D) MEAN	40.92		40.28	41.74	40.44	40.36	41.06	42.24	40.28	40.60	40.40	
	SD	0.18		0.36	0.17	0.25	0.41	0.22	0.22	0.30	0.20	0.17	
	(E) MEAN	39.00		40.60	40.60	39.80	39.00	39.00	40.40	38.60	36.80	36.80	
	SD	0.71		0.89	0.55	0.45	0.71	0.71	0.89	1.14	0.45	0.45	
DDW/H2SO4 + 10 ug/L	(A) MEAN	9.22			11.56	11.50	9.29	10.42	10.74	10.68	11.00	8.40	
	SD	0.79			1.32	0.77	0.57	0.20	0.20	0.69	0.33	0.59	
	(B) MEAN				12.60	10.80	12.50	7.90	10.70	9.90	12.30	9.70	
	SD				1.43	1.03	1.58	0.76	0.67	0.85	1.14	1.36	
	(C) MEAN	10.76	10.20	9.37	9.45	10.54	8.94	9.23	9.65	9.00	9.40	9.95	10.60
	SD	0.22	0.33	0.35	0.30	0.27	0.34	0.20	0.66	0.56	0.44	0.72	0.61
	(D) MEAN	10.12		10.08	10.44	10.00	10.00	10.14	10.30	10.00	9.88	9.72	
	SD	0.11		0.18	0.09	0.00	0.14	0.09	0.10	0.07	0.11	0.20	
	(E) MEAN	9.60		10.00	9.80	10.40	9.80	9.80	10.00	9.60	8.80	9.80	
	SD	0.89		0.07	0.45	2.80	0.45	0.45	0.71	0.89	0.45	0.45	

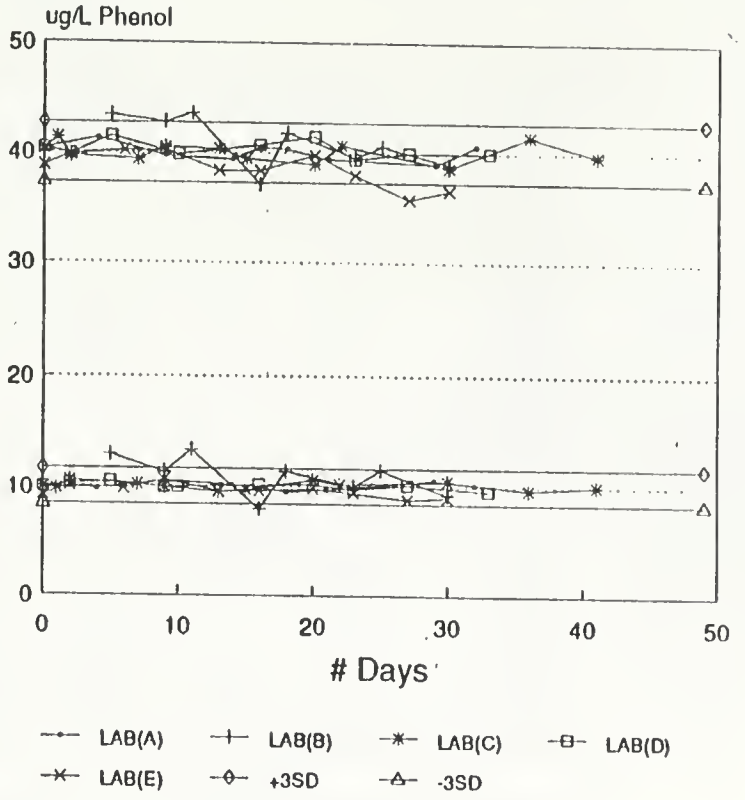
STABILITY STUDY: 4AAP PHENOLICS
PHASE ONE

Figure 1A
Phenol Preserved with H2S04



Stability Study: 4AAP Phenolics

Figure 1B
Phenol Preserved with CuSO4/H3PO4



Stability Study: 4AAP Phenolics

TABLE 2 PHENOLIC SPIKES IN EFFLUENT/ PRESERVED

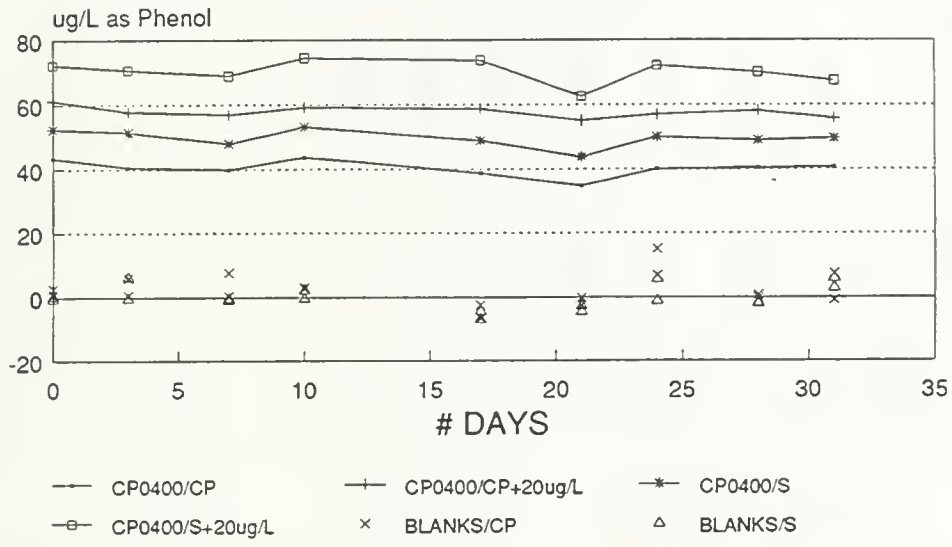
LAB A (EFFLUENT = CONTROL POINT 0400, WEST BAYFRONT SEWER)

DATE / # DAYS		1101 00	1105 03	1108 07	1111 10	1117 17	1122 21	1125 24	1129 28	1202 31
0400/CuSO4	A1	43.81	40.16	39.71	44.83	39.10	35.64	39.76	40.34	40.10
H3PO4	A1	42.93	40.09	39.64	42.80	40.75	36.06	40.82	41.41	39.49
	A2	44.07	40.12	40.77	45.24	37.57	34.51	39.65	41.10	43.31
	A2	44.52	40.38	39.60	45.42	39.82	35.85	43.98	40.75	39.70
	A3	44.15	42.83	40.33	43.02	39.35	32.54	38.55	38.79	41.11
	A3	42.00	40.20	39.38	42.25	36.42	34.65	37.74	40.07	40.57
	MEAN	43.58	40.63	39.91	43.93	38.84	34.88	40.08	40.41	40.71
	SD	0.86	0.99	0.48	1.27	1.44	1.20	1.99	0.85	1.28
0400/CuSO4	C1	62.43	58.11	55.92	59.24	60.23	56.75	57.64	57.80	55.63
H3PO4	C1	60.65	62.00	56.76	61.82	57.62	54.74	58.99	56.80	55.63
+ 20 ug/L	C2	60.17	56.00	57.65	57.65	57.47	55.48	56.79	57.38	55.95
	C2	61.98	55.04	56.87	59.27	57.26	53.86	57.64		56.49
	C3	61.32	55.93	57.09	60.38	58.15	53.97	56.53	62.30	55.63
	C3	61.28	60.48	57.82	57.46	62.09	55.59	54.63	56.73	55.23
	MEAN	61.30	57.93	57.02	59.30	58.80	55.07	57.04	58.20	55.76
	SD	0.76	2.56	0.62	1.51	1.77	1.00	1.33	2.09	0.39
	SPK	17.72	17.30	17.11	15.38	19.97	20.19	16.95	17.79	15.05
DDW/CuSO4	E1	2.59	6.00	7.65	2.62	-2.50	-0.28	15.01	0.62	7.35
H3PO4	E1	1.04	0.56	0.36	3.10	-6.58	-2.53	6.83	-0.27	-0.86
DDW/CuSO4	G1	19.95	17.55	17.92	17.36	14.51	16.01	16.89	16.53	18.09
H3PO4	G1	19.76	18.14	18.62	22.67	15.41	16.08	14.39	17.80	19.64
+ 20 ug/L										
0400/H2SO4	B1	47.15	41.09	43.21	48.93	42.03	39.33	44.27	43.74	45.04
	B1	51.40	48.79	47.87	54.91	47.07		50.70	47.04	53.29
	B2	53.07	51.49	48.75	55.03	50.11	44.36	52.97	48.04	51.31
	B2	54.66	54.15	48.53	54.29	49.50	43.63	50.18	49.45	48.82
	B3	54.36	57.89	49.55	54.62		46.19	51.14	51.16	50.08
	B3	54.29	56.08	50.31	51.70	55.97	46.02	51.32	54.64	49.76
	MEAN	52.49	51.58	48.04	53.25	48.94	43.91	50.10	49.01	49.72
	SD	2.63	5.55	2.29	2.23	4.52	2.49	2.74	3.39	2.52
0400/H2SO4	D1	70.09	64.81	62.62	67.36	69.70	57.28	64.72	62.23	59.88
+ 20 ug/L	D1	70.90	70.03	69.40	74.41	78.63	63.05	69.86	70.34	66.11
	D2	71.13	74.18	69.37	74.12	79.28	65.65	71.92	71.37	69.90
	D2	72.64	72.14	69.26	75.89	73.70	61.46	78.52	74.39	69.03
	D3	74.60	71.29	71.66	77.74	69.59	63.40	75.44	69.99	69.25
	D3	74.09	71.40	71.26	77.52	71.59	64.42		71.51	70.47
	MEAN	72.24	70.64	68.93	74.51	73.75	62.54	72.09	69.97	67.44
	SD	1.67	2.89	2.98	3.48	3.93	2.68	4.73	3.74	3.65
	SPK	19.75	19.06	20.89	21.26	24.81	18.64	22.00	20.96	17.72
0400/H2SO4	F1	-0.52	-0.33	-0.33	2.33	-6.90	-4.43	5.65	-1.65	5.94
	F1	0.33	6.29	-0.91	-0.33	-4.29	-2.74	-1.03	-2.06	2.95
0400/H2SO4	H1	18.84	18.84	16.83	20.09	14.87	15.90	13.36	18.18	17.62
+ 20 ug/L	H1	19.91	18.80	20.11	18.58	16.55	18.79	10.87	18.80	16.75

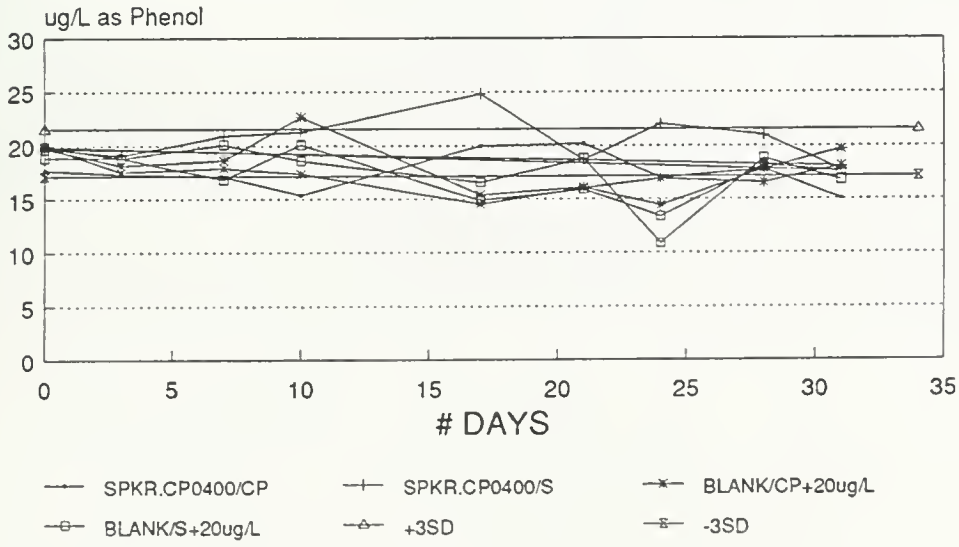
STABILITY STUDY: 4AAP PHENOLICS
PHASE TWO

Figure 2

LAB.A [CP0400, CP0400+Spike]



LAB.A [Spike Recoveries, Spiked Blanks]



Stability Study: 4AAP Phenolics

TABLE 3 PHENOLIC SPIKES IN EFFLUENT/ PRESERVED

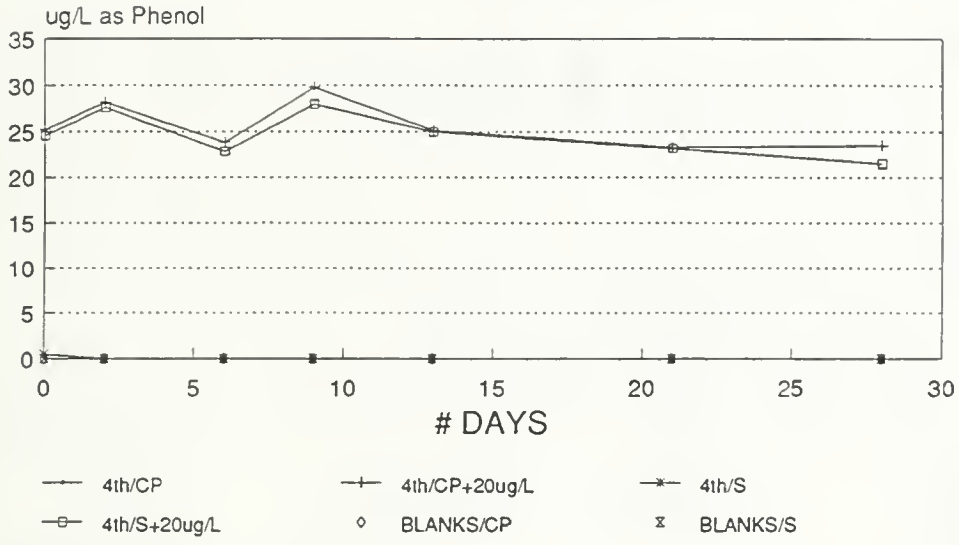
LAB B (EFFLUENT = 4th STREET OUTFALL)

DATE # DAYS		0107 00	0109 02	0113 06	0116 09	0120 13	0128 21	0204 28
4thST/CuSO4	A1	0.00	0.00	0.00	0.00	0.00	0.00	0.00
H3PO4	A1	0.00	0.00	0.00	0.00	0.00	0.00	0.00
	A2	2.44	0.00	0.00	0.00	0.00	0.00	0.00
	A2	0.00	0.00	0.00	0.00	0.00	0.00	0.00
	A3	0.00	0.00	0.00	0.00	0.00	0.00	0.00
	A3	0.00	0.00	0.00	0.00	0.00	0.00	0.00
	MEAN	0.41	0.00	0.00	0.00	0.00	0.00	0.00
	SD	0.91	0.00	0.00	0.00	0.00	0.00	0.00
4thST/CuSO4	C1	26.10	29.30	25.00	32.00	26.00	22.00	23.00
H3PO4	C1	26.00	29.00	24.00	30.00	28.00	24.00	23.00
+ 20 ug/L	C2	23.70	28.00	24.00	30.00	22.00	22.00	21.00
	C2	24.00	25.00	23.00	28.00	25.00	22.00	23.00
	C3	25.60	30.00	24.00	27.00	25.00	25.00	25.00
	C3	26.00	28.00	23.00	32.00	25.00	25.00	26.00
	MEAN	25.23	28.22	23.83	29.83	25.17	23.33	23.50
	SD	0.99	1.60	0.69	1.86	1.77	1.37	1.61
	SPK	24.83	28.22	23.83	29.83	25.17	23.33	23.50
DDW/CuSO4	E1	0.00	0.00	0.00	0.00	0.00	0.00	0.00
H3PO4	E1	0.00	0.00	0.00	0.00	0.00	0.00	0.00
DDW/CuSO4	G1	25.20	29.00	23.00	30.00	27.00	25.00	24.00
H3PO4	G1	25.00	28.00	23.00	28.00	25.00	23.00	19.00
+ 20 ug/L								
4thST/H2SO4	B1	0.00	0.00	0.00	0.00	0.00	0.00	0.00
	B1	0.00	0.00	0.00	0.00	0.00	0.00	0.00
	B2	0.00	0.00	0.00	0.00	0.00	0.00	0.00
	B2	0.00	0.00	0.00	0.00	0.00	0.00	0.00
	B3	0.00	0.00	0.00	0.00	0.00	0.00	0.00
	B3	3.00	0.00	0.00	0.00	0.00	0.00	0.00
	MEAN	0.50	0.00	0.00	0.00	0.00	0.00	0.00
	SD	1.12	0.00	0.00	0.00	0.00	0.00	0.00
4thST/H2SO4	D1	26.00	29.10	23.00	32.00	26.00	24.00	18.00
+ 20 ug/L	D1	27.00	29.00	26.00	31.00	28.00	24.00	24.00
	D2	20.70	26.80	20.00	29.00	23.00	23.00	19.00
	D2	25.00	27.00	22.00	26.00	26.00	22.00	28.00
	D3	24.80	27.00	24.00	29.00	25.00	24.00	20.00
	D3	24.00	27.00	22.00	21.00	22.00	22.00	20.00
	MEAN	24.58	27.65	22.83	28.00	25.00	23.17	21.50
	SD	1.98	0.99	1.86	3.65	2.00	0.90	3.45
	SPK	24.08	27.65	22.83	28.00	25.00	23.17	21.50
DDW/H2SO4	F1	0.00	0.00	0.00	0.00	0.00	0.00	0.00
	F1	0.00	0.00	0.00	0.00	0.00	0.00	0.00
DDW/H2SO4	H1	27.50	32.00	22.00	28.00	27.00	25.00	26.00
+ 20 ug/L	H1	27.00	31.00	23.00	30.00	29.00	25.00	25.00

STABILITY STUDY: 4AAP PHENOLICS
PHASE TWO

Figure 3

LAB.B [4th Str., 4th Str + Spike]



LAB.B [Spike Recoveries, Spiked Blanks]

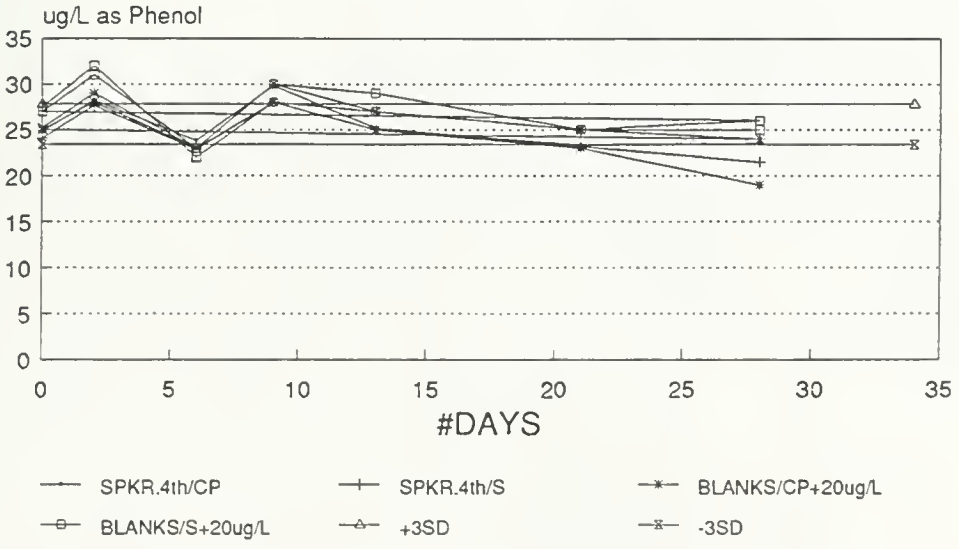


TABLE 4 PHENOLIC SPIKES IN EFFLUENT/ PRESERVED

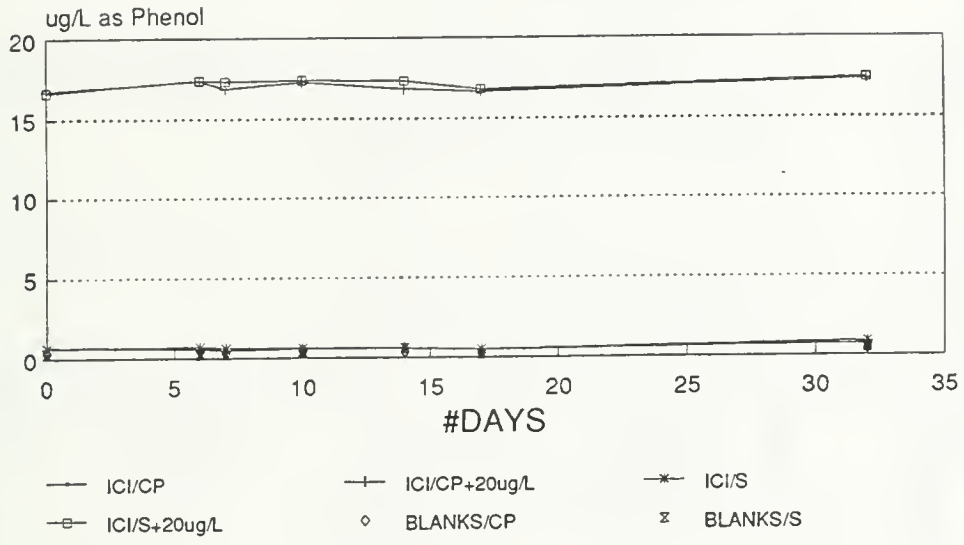
LAB D (EFFLUENT = ICI/CORNWALL)

DATE # DAYS		0228 00	0305 06	0306 07	0309 10	0313 14	0316 17	0331 32
ICI/CuSO4	A1	0.70	0.50	0.50	0.60	0.60	0.55	0.60
H3PO4	A1	0.70	0.70	0.50	0.60	0.55	0.50	0.60
	A2	0.70	0.50	0.50	0.70	0.50	0.50	0.70
	A2	0.70	0.50	0.40	0.65	0.60	0.45	0.70
	A3	0.70	0.60	0.50	0.60	0.60	0.46	0.80
	A3	0.65	0.70	0.50	0.50	0.60	0.50	1.00
	MEAN	0.69	0.58	0.48	0.61	0.58	0.49	0.73
	SD	0.02	0.09	0.04	0.06	0.04	0.03	0.14
ICI/CuSO4	C1	16.85	17.50	16.90	17.50	16.90	16.80	17.50
H3PO4	C1	16.75	17.40	16.90	17.10	16.60	16.70	17.30
+ 20 ug/L	C2	16.75	17.30	17.00	17.30	17.00	16.90	17.60
	C2	16.85	17.20	16.90	17.10	16.80	16.50	17.10
	C3	16.60	17.50	16.80	17.50	16.90	16.70	
	C3	16.85	17.30	16.90	17.20	16.90	16.50	
	MEAN	16.78	17.37	16.90	17.28	16.85	16.68	17.38
	SD	0.09	0.11	0.06	0.17	0.13	0.15	0.19
	SPK	16.08	16.78	16.42	16.67	16.28	16.19	16.64
DDW/CuSO4	E1	0.40	0.40	0.30	0.40	0.40	0.30	0.40
H3PO4	E1	0.30	0.40	0.30	0.50	0.30	0.25	0.60
DDW/CuSO4	G1		21.00	20.70	21.00	20.50	20.40	21.00
H3PO4	G1		20.60	20.90	21.20	20.30	20.30	21.00
+ 20 ug/L								
ICI/H2SO4	B1	0.55	0.70	0.50	0.60	0.60	0.50	0.80
	B1	0.65	0.70	0.50	0.60	0.90	0.50	0.70
	B2	0.70	0.80	0.60	0.70	0.60	0.50	0.90
	B2	0.65	0.70	0.70	0.60	0.55	0.50	1.05
	B3	0.65	0.60	0.70	0.60	0.50	0.55	0.90
	B3	0.65	0.70	0.70	0.60	0.50	0.50	0.90
	MEAN	0.64	0.70	0.62	0.62	0.61	0.51	0.88
	SD	0.04	0.06	0.09	0.04	0.14	0.02	0.11
ICI/H2SO4	D1	16.75	17.10	17.10	17.10	17.10	16.80	17.60
+ 20 ug/L	D1	16.65	17.80	17.32	17.80	17.30	16.75	17.30
	D2	16.80	17.20	17.30	17.20	17.10	16.80	17.20
	D2	16.50	17.20	17.30	17.20	17.00	16.70	17.50
	D3	16.85	17.50	17.50	17.50	16.70	17.10	17.70
	D3	16.50	17.60	17.50	17.60	17.00	16.70	17.50
	MEAN	16.67	17.40	17.34	17.40	17.34	16.81	17.47
	SD	0.14	0.25	0.14	0.25	0.14	0.14	0.17
	SPK	16.03	16.70	16.72	16.78	16.73	16.30	16.59
DDW/H2SO4	F1	0.25	0.20	0.20	0.30	0.60	0.20	0.45
	F1	0.25	0.30	0.20	0.40	0.60	0.25	0.50
DDW/H2SO4	H1		20.90	21.10	21.30	21.00	20.30	20.70
+ 20 ug/L	H1		21.40	21.20	20.70	20.60	20.50	21.10

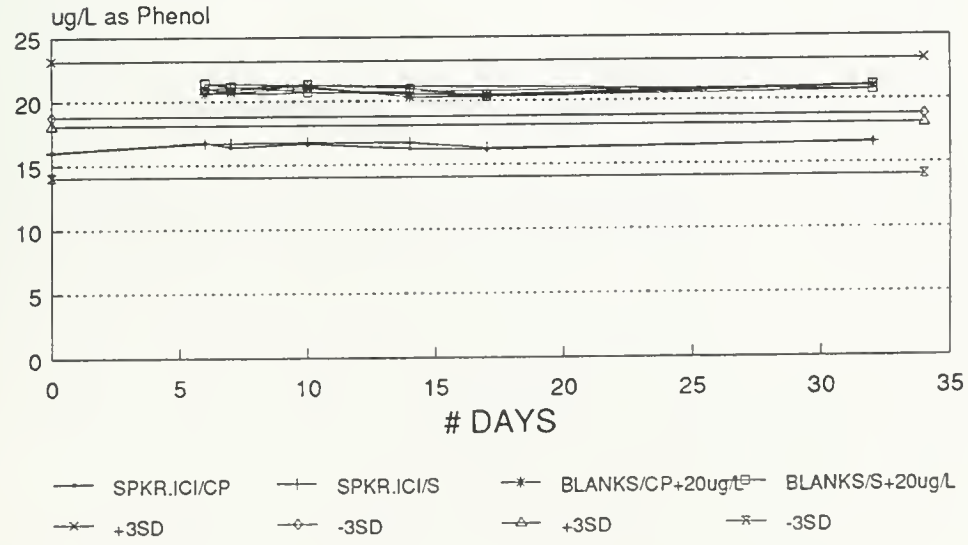
STABILITY STUDY: 4AAP PHENOLICS
PHASE TWO

Figure 4

LAB.D [ICI, ICI+Spike]



LAB.D [Sike Recovery, Spiked Blanks]



Stability Study: 4AAP Phenolics

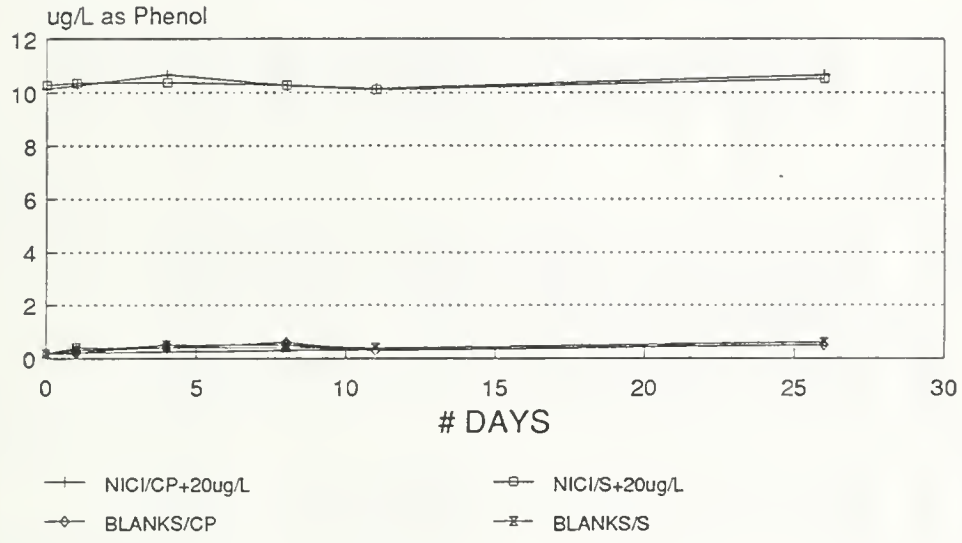
TABLE 5 PHENOLIC SPIKES IN EFFLUENT/ PRESERVED

LAB D (EFFLUENT = ICI/SARNIA)

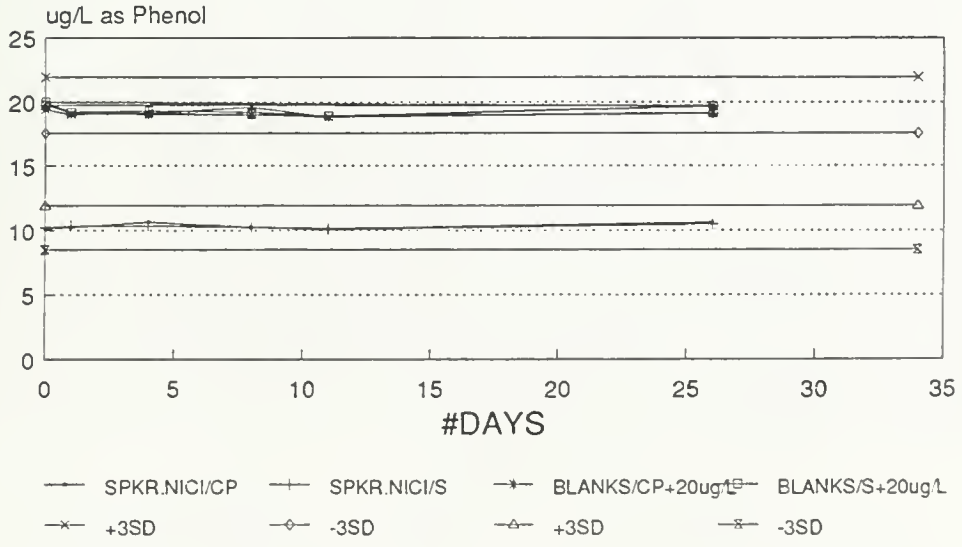
DATE / # DAYS		0228	0305 00	0306 01	0309 04	0313 08	0316 11	0331 26	
NICI/CuSO4	NC1		10.00	10.20	10.70	10.50	10.40	10.60	EFFLUENT UNSPIKED SHOWED
H3PO4	NC1		10.10	10.30	10.70	10.10	10.00	10.70	NO RESPONSE.
+ 20 ug/L	NC2		10.40	10.20	10.75	10.20	10.30	10.60	
	NC2		10.10	10.30	10.70	10.40	10.00	10.60	
	NC3		10.10	10.30	10.55	10.20	10.30	10.90	
	NC3		10.30	10.30	10.60	10.20	10.00	10.50	
	MEAN		10.17	10.27	10.67	10.27	10.17	10.65	
	SD		0.14	0.05	0.07	0.14	0.17	0.13	
NICI/CuSO4	NE1		0.20	0.30	0.40	0.60	0.30	0.50	
H3PO4	NE1		0.20	0.20	0.50	0.50	0.30	0.50	
NICI/CuSO4	NG1		19.40	19.00	19.10	19.60	18.80	19.70	
H3PO4	NG1		19.80	19.10	19.00	19.00	18.80	19.10	
+ 20 ug/L									
DDW/H2SO4	ND1		10.20	10.30	10.70	10.10	10.00	10.50	EFFLUENT UNSPIKED SHOWED
+ 20 ug/L	ND1		10.30	10.40	10.55	10.40	10.00	10.60	NO RESPONSE.
	ND2		10.40	10.35	10.00	10.10	10.10	10.50	
	ND2		10.10	10.30	10.30	10.40	10.10	10.50	
	ND3		10.20	10.30	10.20	10.30	10.20	10.50	
	ND3		10.50	10.50	10.50	10.40	10.30	10.50	
	MEAN		10.28	10.36	10.38	10.28	10.12	10.52	
	SD		0.13	0.07	0.23	0.13	0.11	0.04	
DDW/H2SO4	NF1		0.20	0.40	0.40	0.40	0.40	0.60	
	NF1		0.20	0.30	0.50	0.50	0.40	0.60	
DDW/H2SO4	NH1		19.80	19.20	19.30	19.20	18.90	19.70	
+ 20 ug/L	NH1		20.00	19.20	19.10	19.00	18.90	19.10	

STABILITY STUDY: 4AAP PHENOLICS
PHASE TWO

Figure 5
LAB.D [NICI, NICI+Spike]



LAB.D [Spike Recoveries, Spiked Blanks]



Stability Study: 4AAP Phenolics

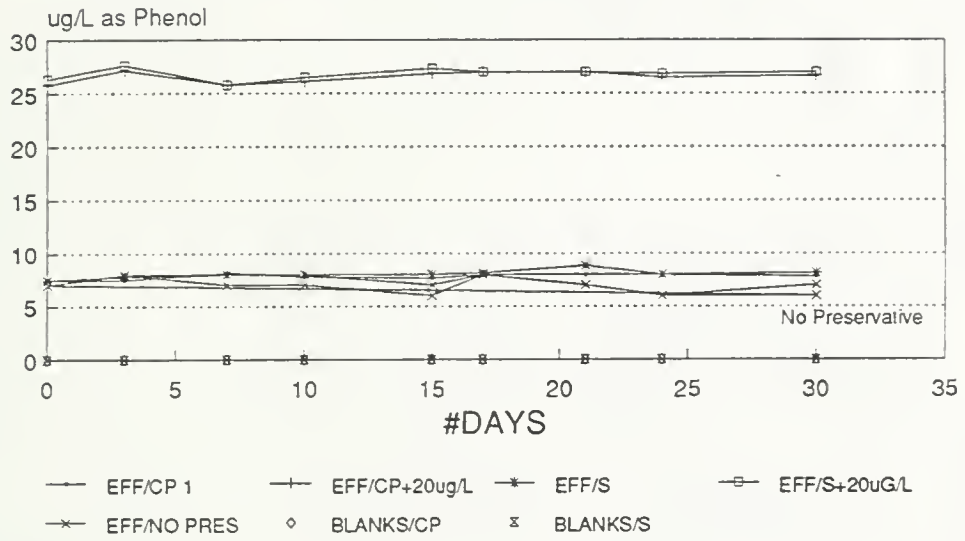
TABLE 6 PHENOLIC SPIKES IN EFFLUENT/ PRESERVED

LAB F (EFFLUENT = CORUNNA SITE EFF)

DATE / # DAYS		1028 00	1031 03	1104 07	1107 10	1112 15	1114 17	1118 21	1121 24	1127 30
EFF/CuSO4 H3PO4	A1	8.00	8.00	9.00	8.00	8.00	8.00	8.00	8.00	8.00
	A1	7.00	8.00	8.00	8.00	8.00	8.00	8.00	8.00	8.00
	A2	7.00	7.00	8.00	8.00	7.00	8.00	8.00	8.00	8.00
	A2	9.00	7.00	8.00	8.00	7.00	8.00	8.00	8.00	8.00
	A3	7.00	7.00	8.00	8.00	8.00	8.00	8.00	8.00	7.00
	A3	7.00	8.00	8.00	7.00	8.00	8.00	8.00	8.00	8.00
	MEAN	7.50	7.50	8.17	7.83	7.67	8.00	8.00	8.00	7.83
	SD	0.76	0.50	0.37	0.37	0.47	0.00	0.00	0.00	0.37
EFF/CuSO4 H3PO4 + 20 ug/L	C1	25.00	27.00	26.00	26.00	27.00	27.00	27.00	27.00	26.00
	C1	26.00	27.00	26.00	27.00	26.00	27.00	27.00	27.00	26.00
	C2	26.00	27.00	26.00	27.00	27.00	27.00	27.00	26.00	27.00
	C2	26.00	27.00	26.00	25.00	27.00	27.00	27.00	26.00	27.00
	C3	26.00	28.00	26.00	25.00	27.00	27.00	27.00	26.00	27.00
	C3	26.00	27.00	25.00	27.00	27.00	27.00	27.00	27.00	27.00
	MEAN	25.83	27.17	25.83	26.17	26.83	27.00	27.00	26.50	26.67
	SD	0.37	0.37	0.37	0.90	0.37	0.00	0.00	0.50	0.47
	SPK	18.33	19.67	17.67	18.33	19.17	19.00	19.00	18.50	18.83
EFF/CuSO4 H3PO4	E1	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
	E1	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
EFF/CuSO4 H3PO4 + 20 ug/L	G1	19.00	20.00	19.00	19.00	20.00	19.00	20.00	19.00	20.00
	G1	19.00	19.00	20.00	20.00	19.00	20.00	19.00	19.00	20.00
EFF/H2SO4	B1	8.00	7.00	8.00	8.00	8.00	8.00	8.00	8.00	8.00
	B1	8.00	8.00	8.00	8.00	8.00	8.00	9.00	8.00	9.00
	B2	7.00	7.00	8.00	8.00	8.00	8.00	9.00	8.00	8.00
	B2	7.00	9.00	8.00	8.00	8.00	8.00	9.00	8.00	8.00
	B3	7.00	8.00	8.00	8.00	8.00	9.00	9.00	8.00	8.00
	B3	8.00	8.00	8.00	8.00	8.00	8.00	9.00	8.00	8.00
	MEAN	7.50	7.83	8.00	8.00	8.00	8.17	8.83	8.00	8.17
	SD	0.50	0.69	0.00	0.00	0.00	0.37	0.37	0.00	0.37
EFF/H2SO4 + 20 ug/L	D1	26.00	28.00	25.00	27.00	27.00	27.00	27.00	27.00	27.00
	D1	27.00	28.00	25.00	26.00	27.00	27.00	27.00	27.00	27.00
	D2	26.00	28.00	25.00	25.00	27.00	27.00	27.00	27.00	27.00
	D2	26.00	27.00	26.00	27.00	27.00	26.00	27.00	26.00	27.00
	D3	27.00	28.00	27.00	27.00	28.00	27.00	27.00	27.00	27.00
	D3	26.00	27.00	27.00	27.00	28.00	28.00	27.00	27.00	27.00
	MEAN	26.33	27.67	25.83	26.50	27.33	27.00	27.00	26.83	27.00
	SD	0.47	0.47	0.90	0.76	0.47	0.58	0.00	0.37	0.00
	SPK	18.83	19.83	17.83	18.50	19.33	18.83	18.17	18.83	18.83
DDW/H2SO4	F1	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
	F1	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
DDW/H2SO4 + 20 ug/L	H1	20.00	20.00	20.00	20.00	20.00	20.00	20.00	19.00	20.00
	H1	19.00	20.00	20.00	20.00	19.00	20.00	19.00	20.00	20.00
EFFLUENT/ NO PRES.	I1	7.00	8.00	7.00	7.00	6.00	8.00	7.00	6.00	6.00
	I2	7.00	8.00	8.00	8.00	7.00	8.00	7.00	6.00	7.00

STABILITY STUDY: 4AAP PHENOLICS
PHASE TWO

Figure 6
LAB.F [EFF, EFF+Spike]



LAB.F [Spike Recoveries, Spiked Blanks]

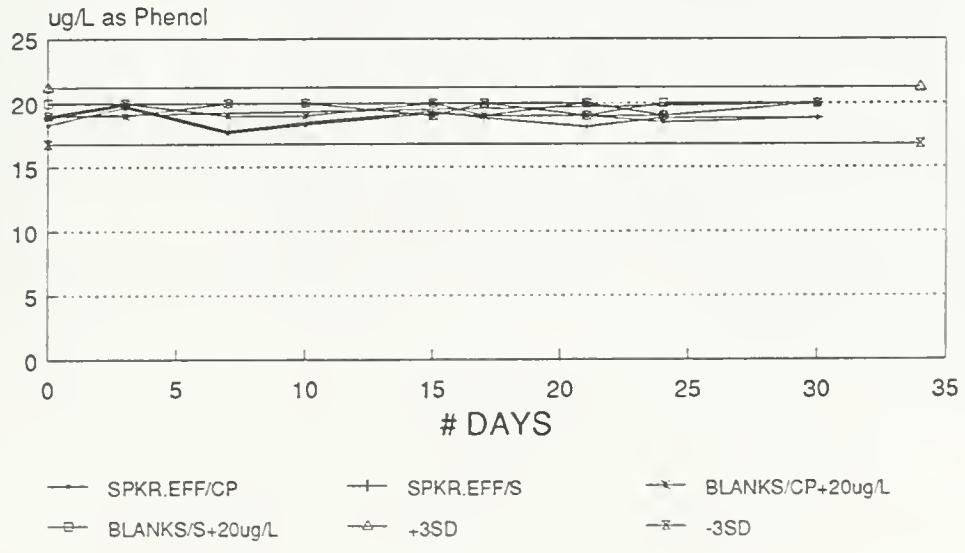


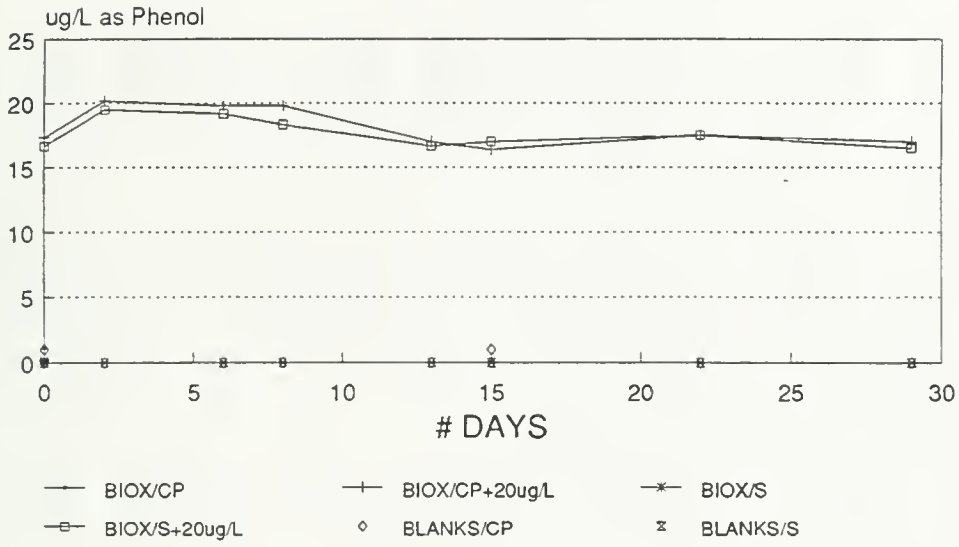
TABLE 7 PHENOLIC SPIKES IN EFFLUENT/ PRESERVED

LAB E (EFFLUENT = BIOX UNIT: CTRL PT 1800)

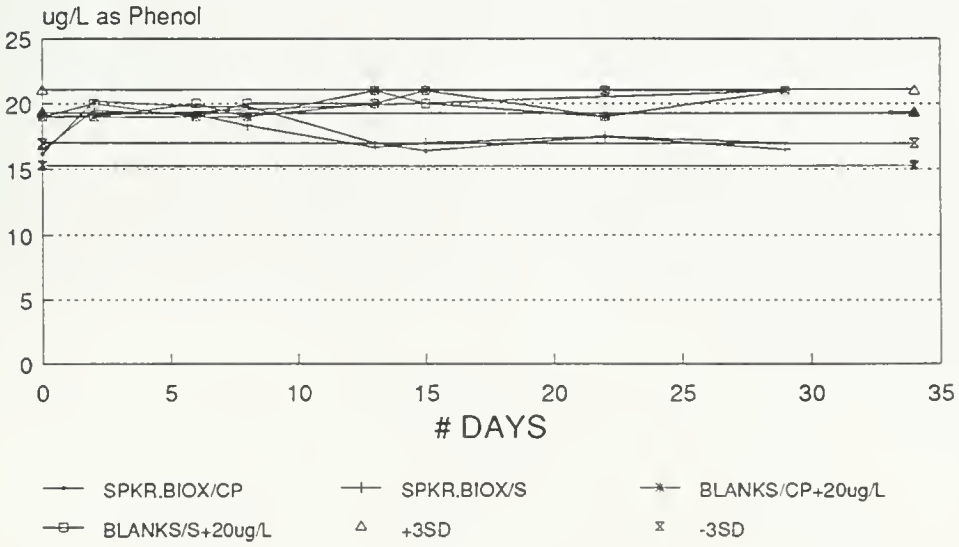
DATE		1023	1025	1029	1031	1105	1107	1114	1121
# DAYS		00	02	06	08	13	15	22	29
BIOX/CuSO4	A1	4.00	NO FURTHER ANALYSIS						
H3PO4	A1	1.00							
	A2	1.00							
	A2	0.00							
	A3	0.00							
	A3	1.00							
	MEAN	1.17							
	SD	1.34							
BIOX/CuSO4	C1	18.00				19.00		16.00	16.00
H3PO4	C1	16.00	20.00	19.00	20.00	14.00	15.00	16.00	17.00
+ 20 ug/L	C2	17.00	20.00	21.00	23.00	17.00	17.00	17.00	17.00
	C2	18.00	21.00	20.00	16.00	17.00	18.00	18.00	16.00
	C3	19.00	21.00	20.00	21.00	18.00	16.00	19.00	17.00
	C3	16.00	19.00	19.00	19.00	17.00	16.00	19.00	19.00
	MEAN	17.33	20.20	19.80	19.80	17.00	16.40	17.50	17.00
	SD	1.11	0.75	0.75	2.32	1.53	1.02	1.26	1.00
	SPK	16.17	20.20	19.80	19.80	17.00	16.40	17.50	17.00
DDW/CuSO4	E1	0.00	0.00	0.00	0.00	0.00	1.00	0.00	0.00
H3PO4	E1	1.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
DDW/CuSO4	G1	19.00	19.00	19.00	19.00	21.00	21.00	19.00	21.00
H3PO4	G1	19.00	19.00	19.00	19.00	20.00	21.00	21.00	21.00
+ 20 ug/L									
BIOX/H2SO4	B1	0.00	NO FURTHER ANALYSIS						
	B1	0.00							
	B2	1.00							
	B2	0.00							
	B3	0.00							
	B3	0.00							
	MEAN	0.17							
	SD	0.37							
BIOX/H2SO4	D1	19.00	20.00	20.00	21.00	16.00	18.00	18.00	16.00
+ 20 ug/L	D1	16.00	20.00	20.00	17.00	16.00	16.00	16.00	16.00
	D2	17.00	20.00	20.00	20.00	17.00	17.00	17.00	17.00
	D2	17.00	19.00	17.00	15.00	18.00	17.00	17.00	16.00
	D3	14.00	20.00	20.00	17.00	17.00	17.00	17.00	16.00
	D3	17.00	18.00	18.00	20.00	16.00	17.00	20.00	18.00
	MEAN	16.67	19.50	19.17	18.33	16.67	17.00	17.50	16.50
	SD	1.49	0.76	1.21	2.13	0.75	0.58	1.26	0.76
	SPK	16.50	19.50	19.17	18.33	16.67	17.00	17.50	16.50
DDW/H2SO4	F1	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
	F1	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
DDW/H2SO4	H1	19.00	19.00	20.00	19.00	21.00	20.00	19.00	21.00
+ 20 ug/L	H1	19.00	20.00	19.00	20.00	20.00	21.00	21.00	21.00

STABILITY STUDY: 4AAP PHENOLICS
PHASE TWO

Figure 7
LAB.E [BIOX, BIOX+Spike]



LAB.E [Spike Recoveries, Spiked Blanks]



Stability Study: 4AAP Phenolics

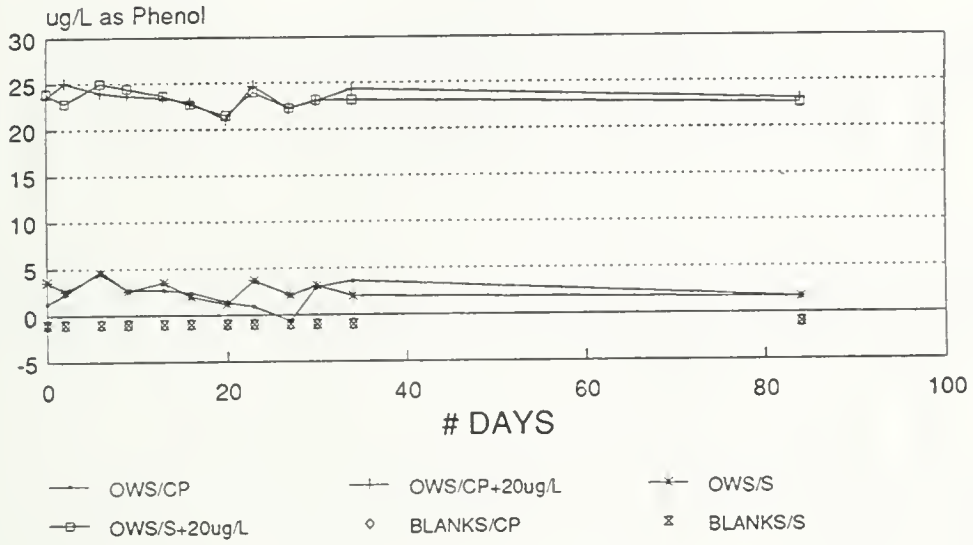
TABLE 8 PHENOLIC SPIKES IN EFFLUENT/ PRESERVED

LAB C (D) (EFFLUENT = OILY WATER SEPARATOR)

DATE / # DAYS		1105 00	1107 02	1111 06	1114 09	1118 13	1121 16	1125 20	1128 23	1202 27	1205 30	1209 34	0128 84
OWS/CuSO4	A1	2.00	-1.00	4.00	2.00	2.50	2.50	1.70	-1.00	-1.00	2.70	2.50	2.00
H3PO4	A1	2.00	3.50	5.00	3.00	3.00	2.00	1.50	-1.00	-1.00	2.70	4.20	2.00
	A2	1.50	3.70	4.50	4.00	2.50	4.00	1.50	1.20	-1.00	3.00	3.50	1.50
	A2	-1.00	2.20	5.00	2.50	2.50	3.00	1.50	2.50	-1.00	3.00	4.50	1.50
	A3	1.50	2.50	5.00	2.00	3.00	1.50	1.00	1.70	-1.00	3.20	3.70	1.50
	A3	1.50	2.50	5.00	2.50	2.50	1.50	1.00	2.00	1.00	3.20	3.50	1.50
	MEAN	1.25	2.23	4.75	2.67	2.67	2.42	1.37	0.90	-0.67	2.97	3.65	1.67
	SD	1.03	1.55	0.38	0.69	0.24	0.89	0.27	1.40	0.75	0.31	0.63	0.24
OWS/CuSO4	C1	22.50	25.00	24.00	22.50	23.00	22.50	23.50	23.50	21.50	22.20	23.50	22.70
H3PO4	C1	24.00	27.00	25.50	23.00	23.00	22.50	21.00	26.50	22.20	22.70	24.00	23.20
+ 20 ug/L	C2	24.00	26.00	23.50	24.50	23.50	23.00	21.50	24.20	21.20	23.00	24.00	22.20
	C2	23.50	25.00	24.00	24.00	23.50	22.50	20.50	24.50	23.00	23.20	24.50	24.00
	C3	24.50	24.00	23.50	24.00	24.00	24.50	20.00	24.50	22.70	23.50	25.20	23.20
	C3	22.50	23.50	23.50	24.00	23.50	22.70	20.00	25.00	23.00	23.70	25.00	23.00
	MEAN	23.50	25.08	24.00	23.67	23.42	22.95	21.08	24.70	22.27	23.05	24.37	23.05
	SD	0.76	1.17	0.71	0.69	0.34	0.72	1.20	0.92	0.71	0.50	0.60	0.55
	SPK	22.25	22.85	19.25	21.00	20.75	20.53	19.72	23.80	22.93	20.08	20.72	21.38
DDW/CuSO4	E1	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00
H3PO4	E1	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00
DDW/CuSO4	G1	20.00	18.50	19.00	21.00	23.00	19.00	20.00	20.20	19.00	20.50	18.20	19.70
H3PO4	G1	20.50	20.00	19.00	23.00	20.00	19.00	20.00	21.70	19.00	20.20	19.70	20.20
+ 20 ug/L													
OWS/H2SO4	B1	4.00	3.50	4.00	2.50	3.00	1.00	2.00	3.50	1.70	3.20	2.00	2.00
	B1	3.50	4.00	4.50	2.50	4.00	2.50	1.50	4.80	2.00	3.50	2.00	2.00
	B2	3.00	1.50	4.50	2.50	3.00	2.00	1.50	5.20	2.00	3.20	2.00	1.50
	B2	2.50	3.00	4.50	2.50	3.00	1.00	1.00	2.70	2.00	3.00	2.00	1.50
	B3	4.00	2.00	5.00	2.50	4.50	3.50	2.00	3.00	3.20	2.70	2.00	1.50
	B3	4.50	1.50	4.50	3.00	3.50	1.70	-1.00	3.00	1.70	3.00	2.00	1.50
	MEAN	3.58	2.58	4.50	2.58	3.50	1.95	1.17	3.70	2.10	3.10	2.00	1.67
	SD	0.67	0.98	0.29	0.19	0.58	0.87	1.03	0.96	0.51	0.24	0.00	0.24
OWS/H2SO4	D1	24.00	22.50	24.50	24.00	23.00	22.50	22.20	23.70	23.00	23.00	23.00	23.20
+ 20 ug/L	D1	24.00	22.50	24.50	24.00	23.50	22.70	21.50	24.70	22.50	23.70	23.70	22.70
	D2	24.00	22.50	26.00	24.50	23.50	23.20	21.00	24.20	22.50	22.00	22.00	22.00
	D2	24.00	23.00	24.50	24.50	24.00	22.00	20.00	23.70	21.70	23.70	23.70	22.00
	D3	24.00	23.00	25.50	25.00	24.00	23.20	23.00	23.70	21.70	23.00	23.00	22.00
	D3	23.50	23.50	25.00	24.50	24.00	22.50	21.20	24.20	22.20	23.50	23.50	23.70
	MEAN	23.92	22.83	25.00	24.42	23.67	22.68	21.48	24.03	22.27	23.15	23.15	22.60
	SD	0.19	0.37	0.58	0.34	0.37	0.42	0.94	0.37	0.46	0.59	0.59	0.67
	SPK	20.33	20.25	20.50	21.83	20.17	20.73	20.32	20.33	20.17	20.05	22.70	20.93
DDW/H2SO4	F1	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00
	F1	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00
DDW/H2SO4	H1	16.50	20.20	20.00	23.00	19.50	19.00	20.00	21.70	19.00	20.70	20.20	19.50
+ 20 ug/L	H1	19.00	19.50	19.00	21.50	24.00	20.00	20.00	20.50	19.50	20.50	22.70	20.20

STABILITY STUDY: 4AAP PHENOLICS
PHASE TWO

Figure 8
LAB.C(D) [OWS, OWS+Spike]



LAB.C(D) [Spike Recoveries, Spiked Blank

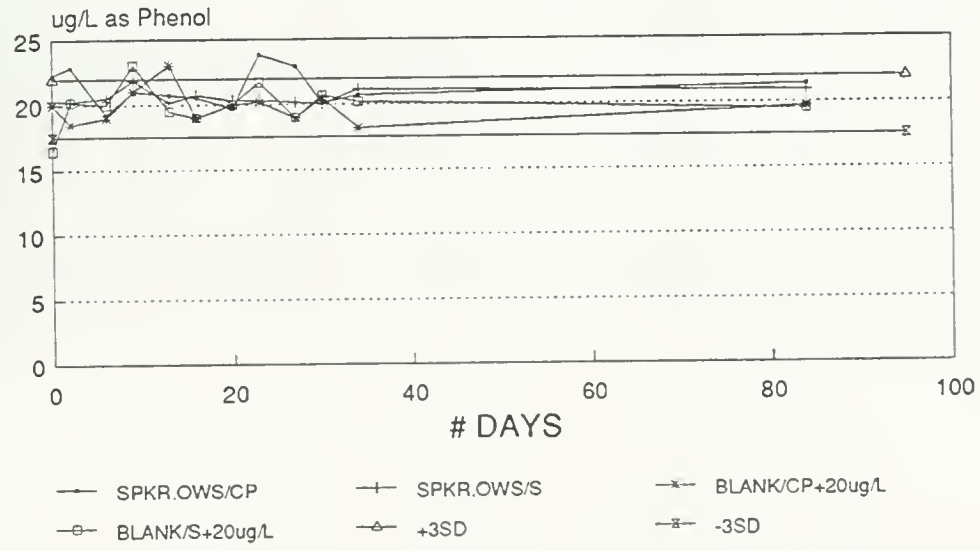


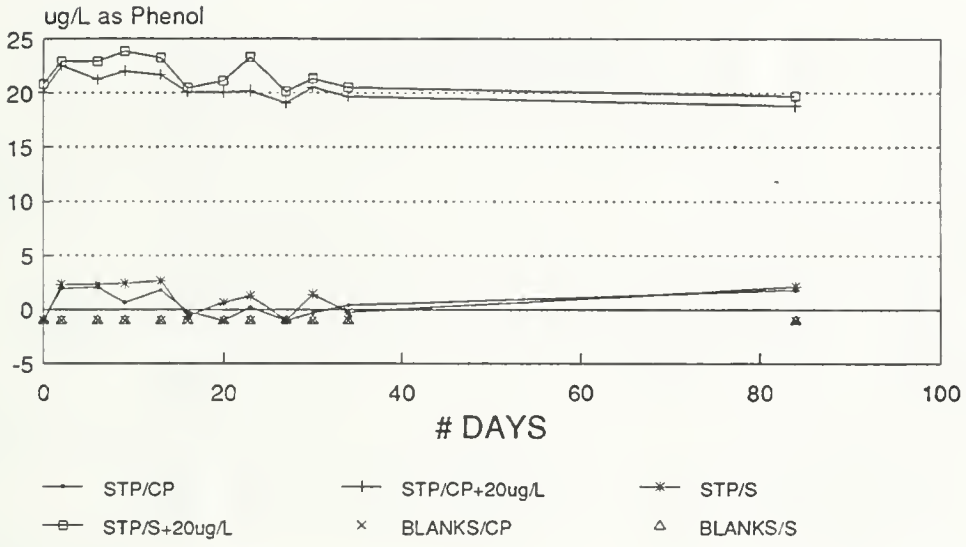
TABLE 9 PHENOLIC SPIKES IN EFFLUENT/ PRESERVED

LAB C (D) (EFFLUENT = SEWAGE TREATMENT PLANT)

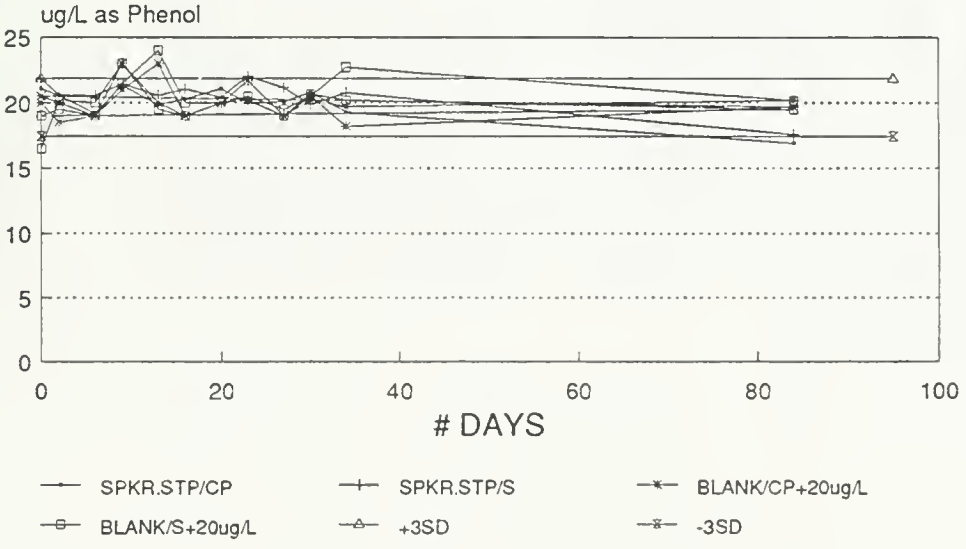
DATE # DAYS		1105 00	1107 02	1111 06	1114 09	1118 13	1121 16	1125 20	1128 23	1202 27	1205 30	1209 34	0128 84
STP/CuSO4 H3P04	A1	-1.00	1.20	2.00	2.00	2.00	-1.00	-1.00	-1.00	-1.00	-1.00	1.20	1.50
	A1	-1.00	2.00	2.00	2.00	1.50	1.00	-1.00	1.20	-1.00	1.20	1.20	1.50
	A2	-1.00	2.00	2.50	-1.00	2.00	-1.00	-1.00	-1.00	-1.00	1.00	1.00	1.70
	A2	-1.00	2.50	2.00	-1.00	1.50	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	2.00
	A3	-1.00	2.00	2.00	1.00	2.00	-1.00	-1.00	1.50	-1.00	-1.00	-1.00	3.00
	A3	-1.00	2.00	2.00	1.00	2.00	2.00	-1.00	1.50	-1.00	-1.00	1.00	1.50
	MEAN	-1.00	1.95	2.08	0.67	1.83	-0.17	-1.00	0.20	-1.00	-0.30	0.40	1.87
	SD	0.00	0.38	0.19	1.25	0.24	1.21	0.00	1.20	0.00	0.99	0.99	0.54
STP/CuSO4 H3P04 + 20 ug/L	C1	20.00	20.00	21.50	20.50	21.00	20.50	20.00	19.70	18.00	20.00	20.20	18.70
	C1	20.50	23.50	22.00	22.50	21.00	20.20	20.00	20.00	19.20	20.70	19.00	18.70
	C2	20.00	22.70	21.50	22.00	22.00	19.50	19.50	19.70	19.00	21.00	20.00	19.00
	C2	19.50	23.00	22.50	22.00	23.00	20.00	20.50	19.70	19.50	20.50	19.20	18.50
	C3	20.50	22.50	20.00	22.00	22.00	20.50	20.50	19.70	19.50	20.50	20.00	19.00
	C3	20.00	23.50	20.00	23.00	21.00	20.00	20.00	22.50	19.20	20.50	19.70	19.00
	MEAN	20.08	22.53	21.25	22.00	21.67	20.12	20.08	20.22	19.07	20.53	19.68	18.82
	SD	0.34	1.19	0.95	0.76	0.75	0.34	0.34	1.03	0.51	0.30	0.44	0.20
	SPK	21.08	20.58	19.17	21.33	19.83	20.28	21.08	20.02	20.07	20.83	19.28	16.95
DDW/CuSO4 H3PO4	E1	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00
	E1	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00
DDW/CuSO4 H3PO4 + 20 ug/L	G1	20.00	18.50	19.00	21.00	23.00	19.00	20.00	20.20	19.00	20.50	18.20	19.70
	G1	20.50	20.00	19.00	23.00	20.00	19.00	20.00	21.70	19.00	20.20	19.70	20.20
STP/H2SO4	B1	-1.00	-1.00	2.00	2.00	2.50	-1.00	-1.00	-1.00	-1.00	1.00	-1.00	1.70
	B1	-1.00	2.00	2.00	2.00	3.50	1.50	-1.00	2.00	-1.00	1.20	-1.00	3.00
	B2	-1.00	3.00	2.00	2.50	2.50	-1.00	1.00	1.50	-1.00	1.50	1.00	2.00
	B2	-1.00	3.70	2.00	3.00	2.50	-1.00	1.00	1.50	-1.00	1.70	-1.00	2.00
	B3	-1.00	3.70	3.00	2.50	2.50	-1.00	2.00	1.50	-1.00	1.70	1.50	2.00
	B3	-1.00	2.50	3.00	2.50	2.50	-1.00	2.00	2.20	-1.00	1.50	1.00	2.20
	MEAN	-1.00	2.32	2.33	2.42	2.67	-0.58	0.67	1.28	-1.00	1.43	-0.25	2.15
	SD	0.00	1.60	0.47	0.34	0.37	0.93	1.25	1.06	0.00	0.26	1.07	0.41
STP/H2SO4 + 20 ug/L	D1	20.00	23.50	22.50	24.00	23.00	20.50	19.00	22.50	19.70	20.50	20.00	18.20
	D1	20.00	23.50	22.50	24.00	23.00	20.50	20.00	22.70	20.00	21.00	20.00	19.50
	D2	20.50	22.50	24.00	24.50	24.50	21.00	22.70	23.50	20.00	22.70	20.00	20.00
	D2	21.50	24.50	23.00	23.50	23.50	19.70	20.50	23.00	20.20	21.00	20.20	19.70
	D3	20.50	21.50	23.00	23.50	22.50	21.00	21.50	24.20	20.50	21.50	21.00	20.50
	D3	22.50	22.00	22.50	23.50	23.00	20.00	23.00	24.00	20.50	21.20	22.00	20.50
	MEAN	20.83	22.92	22.92	23.83	23.25	20.45	21.12	23.32	20.15	21.32	20.53	19.73
	SD	0.90	1.02	0.53	0.37	0.63	0.48	1.43	0.64	0.29	0.69	0.75	0.78
	SPK	21.83	20.60	20.58	21.42	20.58	21.03	20.45	22.03	21.15	19.88	20.78	17.58
DDW/H2SO4	F1	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00
	F1	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00
DDW/H2SO4 + 20 ug/L	H1	16.50	20.20	20.00	23.00	19.50	19.00	20.00	21.70	19.00	20.70	20.20	19.50
	H1	19.00	19.50	19.00	21.50	24.00	20.00	20.00	20.50	19.50	20.50	20.70	20.20

STABILITY STUDY: 4AAP PHENOLICS
PHASE TWO

Figure 9
LAB.C(D) [STP, STP+Spike]



LAB.C(D) [Spike Recoveries, Spiked Blank



Stability Study: 4AAP Phenolics

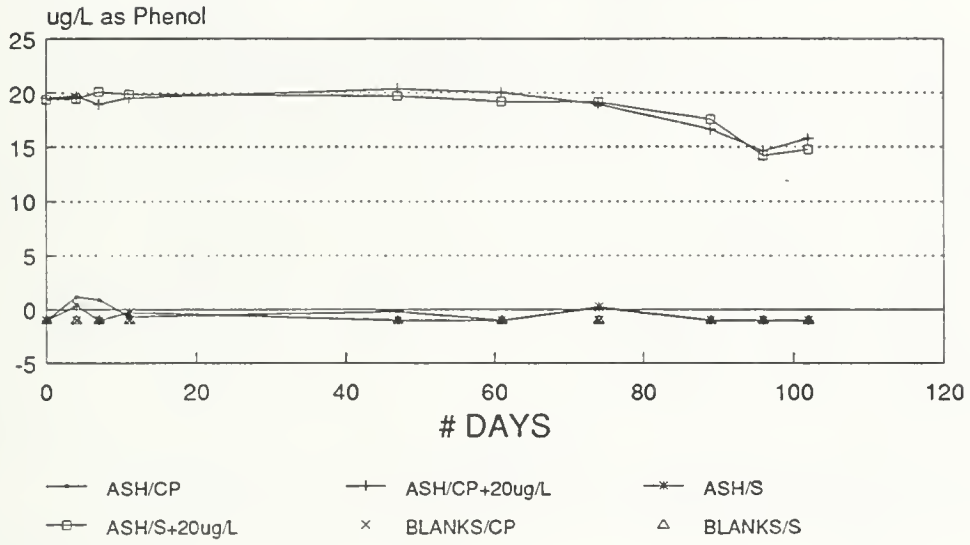
TABLE 10 PHENOLIC SPIKES IN EFFLUENT/ PRESERVED

LAB C (N) (EFFLUENT = ASH LAGOON)

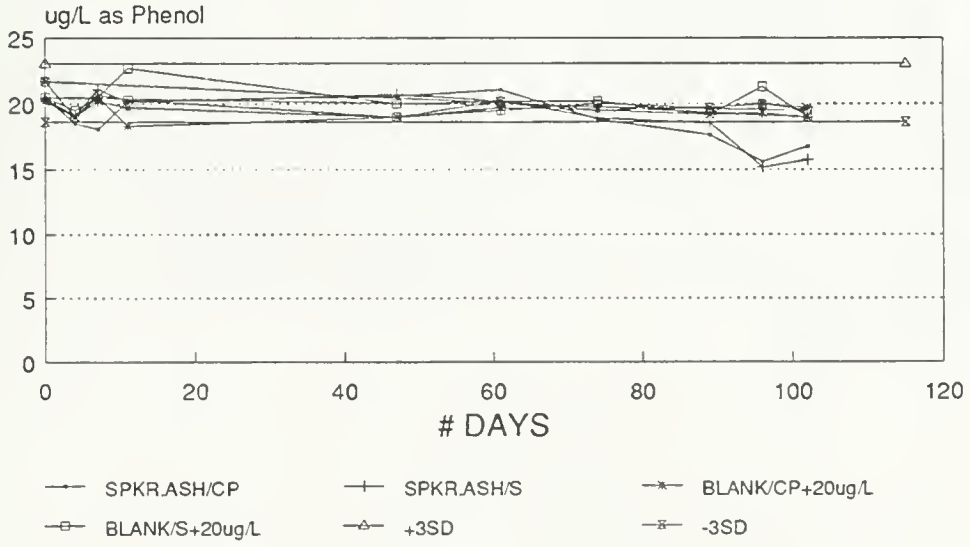
DATE / # DAYS		1128 00	1202 04	1205 07	1209 11	0114 47	0128 61	0210 74	0225 89	0303 96	0309 102
ASH/CuSO4 H3PO4	A1	-1.00	1.00	1.20	1.00	-1.00	-1.00	1.00	-1.00	-1.00	-1.00
	A1	-1.00	1.20	1.00	-1.00	-1.00	-1.00	1.20	-1.00	-1.00	-1.00
	A2	-1.00	2.00	1.50	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00
	A2	-1.00	2.00	1.50	-1.00	1.70	-1.00	-1.00	-1.00	-1.00	-1.00
	A3	-1.00	2.00	1.20	-1.00	1.20	-1.00	-1.00	-1.00	-1.00	-1.00
	A3	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	1.70	-1.00	-1.00	-1.00
	MEAN	-1.00	1.20	0.90	-0.67	-0.18	-1.00	0.15	-1.00	-1.00	-1.00
	SD	0.00	1.06	0.87	0.75	1.16	0.00	1.17	0.00	0.00	0.00
ASH/CuSO4 H3PO4 +20 ug/L	C1	19.20	18.50	18.20	19.50	19.50	20.00	19.00	16.00	14.00	17.00
	C1	19.00	19.20	19.00	20.00	21.00	21.00	19.20	17.00	13.20	16.00
	C2	19.50	22.00	19.00	19.50	20.70	20.00	19.00	17.20	15.00	17.50
	C2	19.50	19.50	19.20	20.00	20.50	20.20	19.20	16.00	14.50	15.20
	C3	20.00	19.50	19.00	18.70	20.20	20.20	19.00	16.70	14.50	16.30
	C3	20.00	19.50	19.50	19.50	20.50	19.00	18.50	17.00	16.50	12.70
	MEAN	19.53	19.70	18.98	19.53	20.40	20.07	18.98	16.65	14.62	15.78
	SD	0.37	1.09	0.39	0.43	0.47	0.58	0.23	0.48	1.01	1.56
	SPK	20.53	18.50	18.08	20.20	20.58	21.07	18.83	17.65	15.62	16.78
ASH/CuSO4 H3PO4	E1	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00
	E1	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00
ASH/CuSO4 H3PO4 +20 ug/L	G1	20.20	19.00	20.50	18.30	19.00	19.70	19.50	19.20	19.30	19.00
	G1	21.70	19.00	20.20	19.70	19.00	20.20	19.50	19.70	20.00	19.70
ASH/H2SO4	B1	-1.00	1.00	-1.00	-1.00	-1.00	-1.00	1.50	-1.00	-1.00	-1.00
	B1	-1.00	-1.00	-1.00	1.20	-1.00	-1.00	1.70	-1.00	-1.00	-1.00
	B2	-1.00	1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00
	B2	-1.00	1.00	-1.00	1.20	-1.00	-1.00	1.20	-1.00	-1.00	-1.00
	B3	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00
	B3	-1.00	1.20	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00
	MEAN	-1.00	0.37	-1.00	-0.27	-1.00	-1.00	0.23	-1.00	-1.00	-1.00
	SD	0.00	0.97	0.00	1.04	0.00	0.00	1.24	0.00	0.00	0.00
ASH/H2SO4 +20 ug/L	D1	17.80	19.70	20.00	19.50	18.50	19.20	19.00	17.00	11.50	15.00
	D1	18.00	18.20	19.70	19.70	19.50	19.20	19.20	17.50	13.50	16.30
	D2	24.20	19.50	20.00	19.70	20.50	19.00	19.00	17.50	13.00	12.20
	D2	18.50	20.00	20.00	20.00	19.70	19.70	19.30	17.70	16.00	14.70
	D3	18.70	20.00	20.70	20.20	20.20	19.00	19.00	17.70	15.20	14.70
	D3	19.20	19.50	20.20	20.20	20.00	19.20	19.50	18.00	16.00	15.70
	MEAN	19.40	19.48	20.10	19.88	19.73	19.22	19.17	17.57	14.20	14.77
	SD	2.19	0.61	0.31	0.27	0.64	0.23	0.19	0.30	1.67	1.28
	SPK	20.40	19.12	21.10	20.15	20.73	20.22	18.93	18.57	15.20	15.77
ASH/H2SO4	F1	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00
	F1	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00
ASH/H2SO4 20 ug/L	H1	21.70	19.00	20.70	20.30	19.00	19.50	20.00	19.70	20.00	19.50
	H1	20.50	19.50	20.50	22.70	20.00	20.20	20.20	19.20	21.30	19.00

STABILITY STUDY: 4AAP PHENOLICS
PHASE TWO

Figure 10
LAB.C(N) [ASH, ASH+Spike]



LAB.C(N) [Spike Recoveries, Spiked Blank



Stability Study: 4AAP Phenolics

TABLE 11 PHENOLIC SPIKES IN EFFLUENT/ PRESERVED

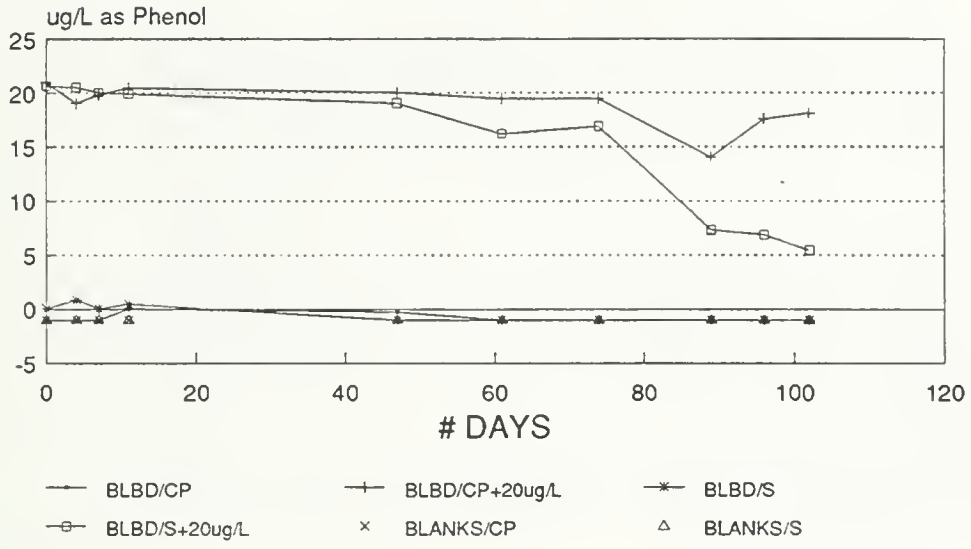
LAB C (N) (EFFLUENT = BOILER BLOWDOWN)

DATE		1128	1202	1205	1209	0114	0128	0210	0225	0303	0309
# DAYS		00	04	07	11	47	61	74	89	96	102
BLBD/CuSO4	A1	-1.00	-1.00	-1.00	-1.00	1.50	-1.00	-1.00	-1.00	-1.00	-1.00
H3PO4	A1	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00
	A2	-1.00	-1.00	-1.00	1.20	1.00	-1.00	-1.00	-1.00	-1.00	-1.00
	A2	-1.00	-1.00	-1.00	1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00
	A3	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00
	A3	-1.00	-1.00	-1.00	1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00
	MEAN	-1.00	-1.00	-1.00	0.03	-0.25	-1.00	-1.00	-1.00	-1.00	-1.00
	SD	0.00	0.00	0.00	1.04	1.07	0.00	0.00	0.00	0.00	0.00
BLBD/CuSO4	C1	20.20	18.50	20.00	19.50	20.20	19.00	19.50	13.50	17.50	18.50
H3PO4	C1	21.20	19.00	20.00	20.50	20.00	20.00	19.00	14.50	18.70	18.00
+20 ug/L	C2	20.20	19.50	20.00	22.70	20.70	19.70	19.50	14.30	17.70	17.20
	C2	21.00	19.20	19.70	19.70	20.20	19.70	19.50	13.70	17.30	18.50
	C3	22.70	19.20	19.20	20.50	20.20	19.50	20.00	14.00	17.50	18.00
	C3	20.50	19.00	20.00	20.00	19.00	19.00	19.50	14.20	17.00	18.70
	MEAN	20.97	19.07	19.82	20.48	20.05	19.48	19.50	14.03	17.62	18.15
	SD	0.86	0.30	0.30	1.06	0.52	0.37	0.29	0.34	0.53	0.50
	SPK	21.97	20.07	20.82	20.45	20.30	20.48	20.50	15.03	18.62	19.15
BLBD/CuSO4	E1	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00
H3PO4	E1	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00
BLBD/CuSO4	G1	20.20	19.00	20.50	18.30	19.00	19.70	19.50	19.20	19.30	19.00
H3PO4	G1	21.70	19.00	20.20	19.70	19.00	20.20	19.50	19.70	20.00	19.70
+20 ug/L											
BLBD/H2SO4	B1	-1.00	1.00	1.20	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00
	B1	1.20	1.20	-1.00	1.20	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00
	B2	1.00	-1.00	1.00	1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00
	B2	1.00	1.20	-1.00	1.50	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00
	B3	-1.00	1.70	1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00
	B3	-1.00	1.00	-1.00	1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00
	MEAN	0.03	0.85	0.03	0.45	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00
	SD	1.04	0.86	1.04	1.04	0.00	0.00	0.00	0.00	0.00	0.00
BLBD/H2SO4	D1	19.70	19.50	19.50	19.50	19.00	15.20	15.50	5.70	4.00	1.30
+20 ug/L	D1	21.70	20.00	20.00	19.50	18.00	14.50	17.20	10.00	5.50	6.50
	D2	20.30	21.00	19.70	20.00	18.20	16.00	17.00	9.20	5.70	3.70
	D2	20.50	20.50	20.00	20.20	21.50	15.70	16.70	5.50	5.70	3.50
	D3	20.50	21.50	20.50	20.50	19.20	18.00	18.50	4.70	4.30	11.00
	D3	21.20	20.70	20.70	19.80	18.50	18.00	16.70	8.70	16.00	6.70
	MEAN	20.65	20.53	20.07	19.92	19.07	16.23	16.93	7.30	6.87	5.45
	SD	0.64	0.65	0.42	0.36	1.17	1.33	0.88	2.06	4.14	3.10
	SPK	20.62	19.68	20.03	19.47	20.07	17.23	17.93	8.30	7.87	6.45
BLBD/H2SO4	F1	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00
	F1	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00
BLBD/H2SO4	H1	21.70	19.00	20.70	20.30	19.00	19.50	20.00	19.70	20.00	19.50
+20 ug/L	H1	20.50	19.50	20.50	22.70	20.00	20.20	20.20	19.20	21.30	19.00

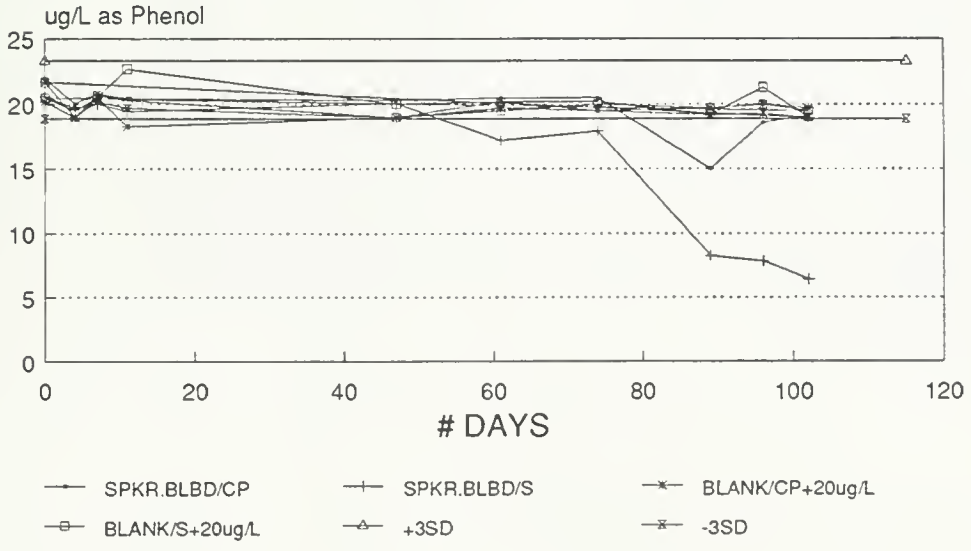
STABILITY STUDY: 4AAP PHENOLICS
PHASE TWO

Figure 11

LAB.C(N) [BLBD, BLBD+Spike]



LAB.C(N) [Spike Recoveries, Spiked Blank



Stability Study: 4AAP Phenolics

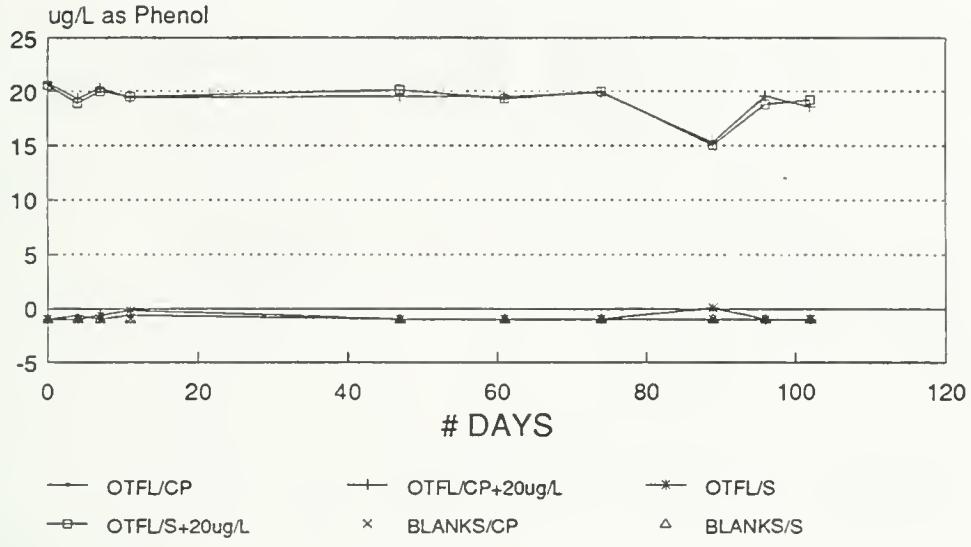
TABLE 12 PHENOLIC SPIKES IN EFFLUENT/ PRESERVED

LAB C (N) (EFFLUENT = OUTFALL)

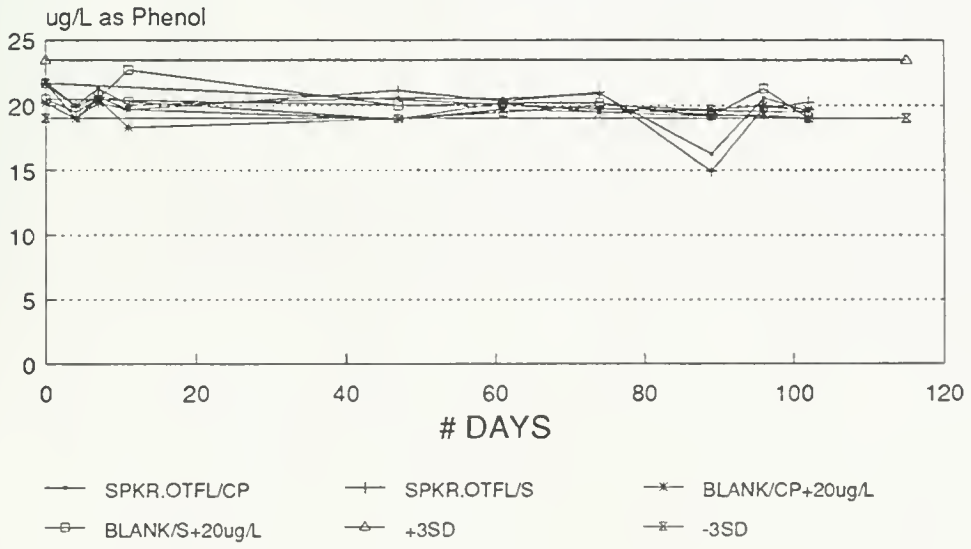
DATE / # DAYS		1128 00	1202 04	1205 07	1209 11	0114 47	0128 61	0210 74	0225 89	0303 96	0309 102
OTFL/CuSO4 H3PO4	A1	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00
	A1	-1.00	1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00
	A2	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00
	A2	-1.00	-1.00	-1.00	1.20	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00
	A3	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00
	A3	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00
	MEAN	-1.00	-0.67	-1.00	-0.63	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00
	SD	0.00	0.75	0.00	0.82	0.00	0.00	0.00	0.00	0.00	0.00
OTFL/CuSO4 H3PO4 +20 ug/L	C1	20.70	18.20	19.50	19.00	19.50	19.00	19.50	14.30	18.50	18.50
	C1	21.00	18.70	20.20	19.50	20.50	19.00	19.70	15.00	19.50	17.70
	C2	20.70	19.70	20.00	20.20	19.20	21.20	20.20	15.50	20.00	18.70
	C2	21.50	19.70	20.20	19.50	20.00	19.70	20.00	15.70	19.50	19.20
	C3	20.50	19.70	21.70	19.20	18.70	19.20	20.50	15.50	20.00	18.50
	C3	20.50	20.00	20.20	19.20	19.50	19.00	19.50	15.70	20.00	19.00
	MEAN	20.82	19.33	20.30	19.43	19.57	19.52	19.90	15.28	19.58	18.60
	SD	0.35	0.65	0.67	0.39	0.57	0.79	0.37	0.50	0.53	0.48
	SPK	21.82	20.00	21.30	20.07	20.57	20.52	20.90	16.28	20.58	19.60
OTFL/CuSO4 H3PO4	E1	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00
	E1	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00
OTFL/CuSO4 H3PO4 +20 ug/L	G1	20.20	19.00	20.50	18.30	19.00	19.70	19.50	19.20	19.30	19.00
	G1	21.70	19.00	20.20	19.70	19.00	20.20	19.50	19.70	20.00	19.70
OTFL/H2SO4	B1	-1.00	-1.00	-1.00	1.50	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00
	B1	-1.00	-1.00	1.20	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00
	B2	-1.00	-1.00	-1.00	1.50	-1.00	-1.00	-1.00	1.00	-1.00	-1.00
	B2	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00
	B3	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	1.00	-1.00	-1.00
	B3	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	1.50	-1.00	-1.00
	MEAN	-1.00	-1.00	-0.63	-0.17	-1.00	-1.00	-1.00	0.08	-1.00	-1.00
	SD	0.00	0.00	0.82	1.18	0.00	0.00	0.00	1.10	0.00	0.00
OTFL/H2SO4 +20 ug/L	D1	19.70	19.50	19.70	19.20	19.00	18.20	19.20	13.50	20.00	18.50
	D1	20.70	19.20	20.00	19.20	20.20	19.20	20.00	15.70	18.50	19.00
	D2	20.50	18.70	20.20	20.00	19.50	20.00	20.20	17.70	18.00	19.20
	D2	21.70	19.50	20.00	19.70	22.20	19.50	19.20	14.50	18.50	19.50
	D3	20.50	18.50	20.20	19.50	19.50	19.50	20.20	14.30	19.00	19.50
	D3	20.50	18.20	20.00	19.50	20.50	19.50	21.20	14.50	19.00	19.70
	MEAN	20.60	18.93	20.02	19.52	20.15	19.32	20.00	15.03	18.83	19.23
	SD	0.59	0.50	0.17	0.28	1.04	0.55	0.68	1.35	0.62	0.40
	SPK	21.60	19.93	20.65	19.68	21.15	20.32	21.00	14.95	19.83	20.23
OTFL/H2SO4	F1	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00
	F1	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00
OTFL/H2SO4 +20 ug/L	H1	21.70	19.00	20.70	20.30	19.00	19.50	20.00	19.70	20.00	19.50
	H1	20.50	19.50	20.50	22.70	20.00	20.20	20.20	19.20	21.30	19.00

STABILITY STUDY: 4AAP PHENOLICS
PHASE TWO

Figure 12
LAB.C(N) [OTFL, 0TFL+Spike]



LAB.C(N) [Spike Recoveries, Spiked Blank



Stability Study: 4AAP Phenolics

TABLE 13 PHENOLIC SPIKES IN EFFLUENT/ PRESERVED

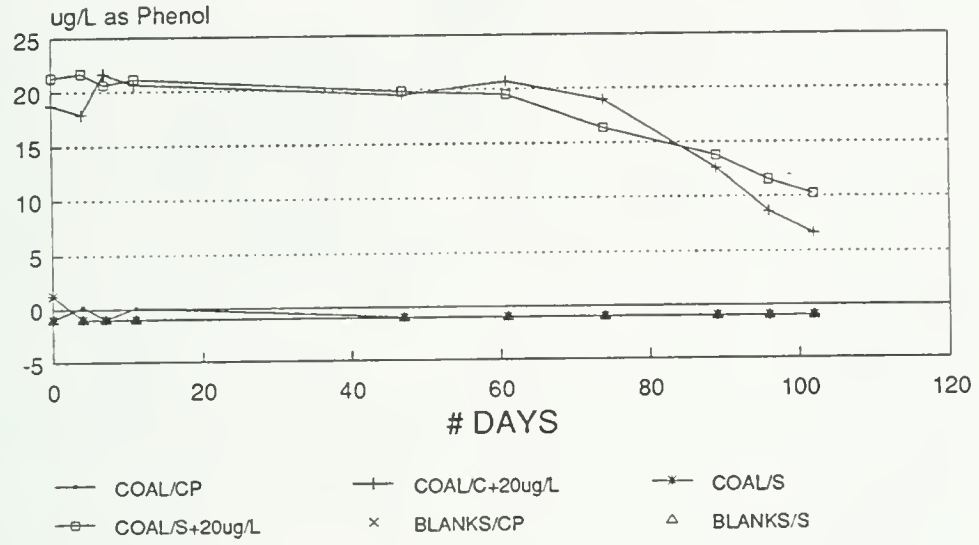
LAB C (N) (EFFLUENT = COAL PILE RUNOFF)

DATE / # DAYS		1128 00	1202 04	1205 07	1209 11	0114 47	0128 61	0210 74	0225 89	0303 96	0309 102
COAL/CuSO4	A1	-1.00	1.50	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00
H3PO4	A1	-1.00	1.20	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00
	A2	-1.00	1.70	-1.00	1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00
	A2	-1.00	-1.00	-1.00	1.20	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00
	A3	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00
	A3	-1.00	-1.00	-1.00	1.50	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00
	MEAN	-1.00	0.23	-1.00	0.12	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00
	SD	0.00	1.24	0.00	1.13	0.00	0.00	0.00	0.00	0.00	0.00
COAL/CuSO4	C1	18.70	18.50	19.70	20.50	20.00	20.00	18.20	10.30	4.70	-1.00
H3PO4	C1	19.20	17.80	20.00	20.00	19.00	20.50	18.50	10.30	5.20	1.30
+20 ug/L	C2	18.70	18.20	29.70	20.70	19.50	20.00	20.00	13.70	10.20	9.00
	C2	19.20	18.50	20.00	21.20	20.00	20.50	18.70	13.00	8.70	9.70
	C3	19.00	17.70	20.50	20.50	19.00	20.20	19.50	14.00	11.00	10.50
	C3	18.00	17.00	20.20	21.20	19.70	23.00	19.00	14.30	12.00	10.30
	MEAN	18.80	17.95	21.68	20.68	19.53	20.70	18.98	12.60	8.63	6.63
	SD	0.41	0.53	3.59	0.42	0.41	1.05	0.61	1.67	2.79	4.66
	SPK	19.80	17.72	22.68	20.57	20.53	21.70	19.98	13.60	9.63	7.63
COAL/CuSO4	E1	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00
H3PO4	E1	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00
COAL/CuSO4	G1	20.20	19.00	20.50	18.30	19.00	19.70	19.50	19.20	19.30	19.00
H3PO4	G1	21.70	19.00	20.20	19.70	19.00	20.20	19.50	19.70	20.00	19.70
+20 ug/L											
COAL/H2SO4	B1	2.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00
	B1	1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00
	B2	1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00
	B2	1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00
	B3	1.50	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00
	B3	1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00
	MEAN	1.25	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00
	SD	0.38	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
COAL/H2SO4	D1	19.50	21.00	20.00	20.70	22.20	20.70	18.00	14.70	14.00	12.20
+20 ug/L	D1	20.50	21.00	20.50	20.70	20.00	20.70	18.00	15.50	14.00	11.70
	D2	23.50	22.00	20.50	20.70	18.70	20.20	17.50	14.20	12.30	12.00
	D2	21.50	21.50	20.50	21.00	20.20	21.00	17.50	14.70	14.00	12.30
	D3	21.50	22.20	21.20	22.00	19.00	17.50	13.20	12.00	7.50	6.50
	D3	21.50	22.70	21.20	22.00	19.00	17.00	14.00	11.50	7.30	6.50
	MEAN	21.33	21.73	20.65	21.18	19.85	19.52	16.37	13.77	11.52	10.20
	SD	1.21	0.63	0.43	0.59	1.19	1.63	1.98	1.48	2.97	2.62
	SPK	20.08	22.73	21.65	22.18	20.85	20.52	17.37	14.77	12.52	11.20
COAL/H2SO4	F1	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00
	F1	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00
COAL/H2SO4	H1	21.70	19.00	20.70	20.30	19.00	19.50	20.00	19.70	20.00	19.50
+20 ug/L	H1	20.50	19.50	20.50	22.70	20.00	20.20	20.20	19.20	21.30	19.00

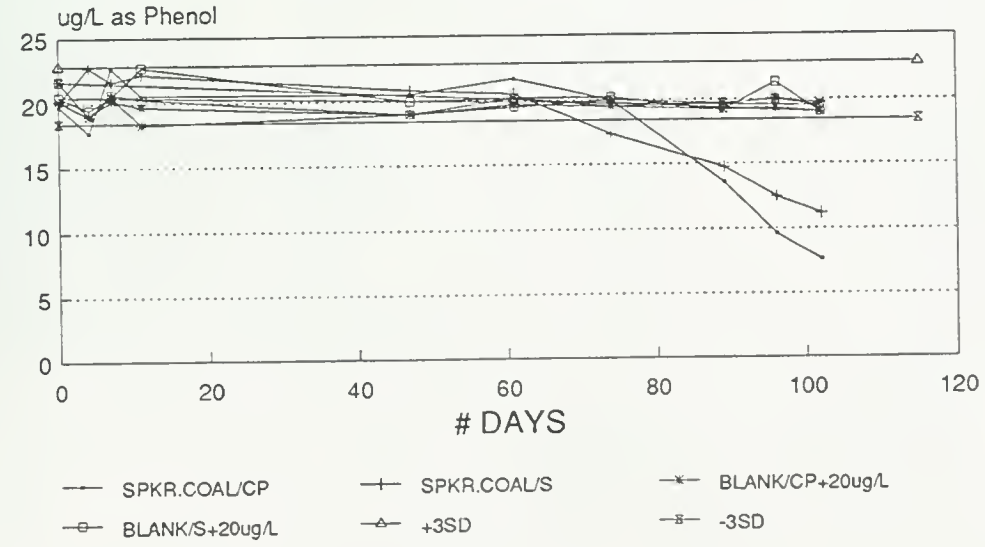
STABILITY STUDY: 4AAP PHENOLICS
PHASE TWO

Figure 13

LAB.C(N) [COAL, COAL+Spike]



LAB.C(N) [Spike Recoveries, Spiked Blank



Stability Study: 4AAP Phenolics

Figure 14A
LABS A to F: Spiked Effluents

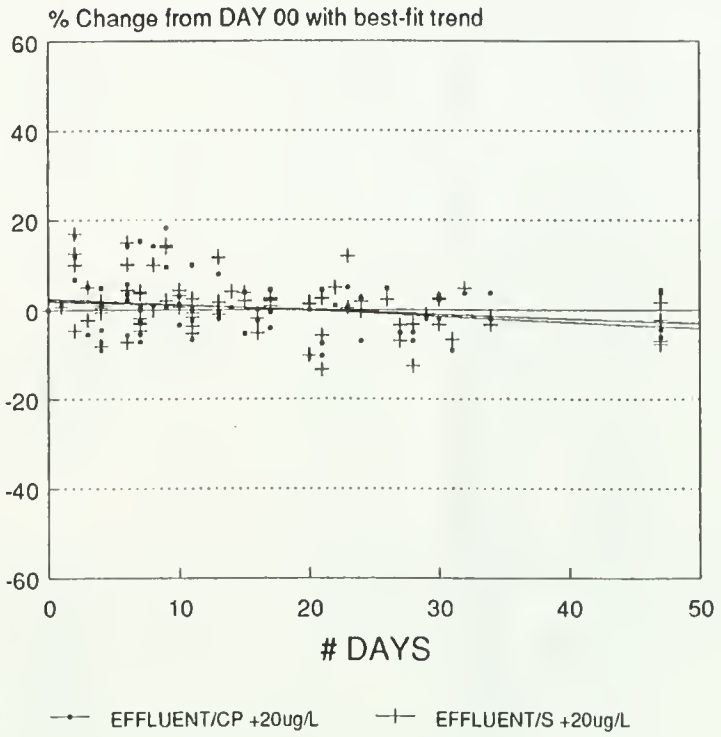
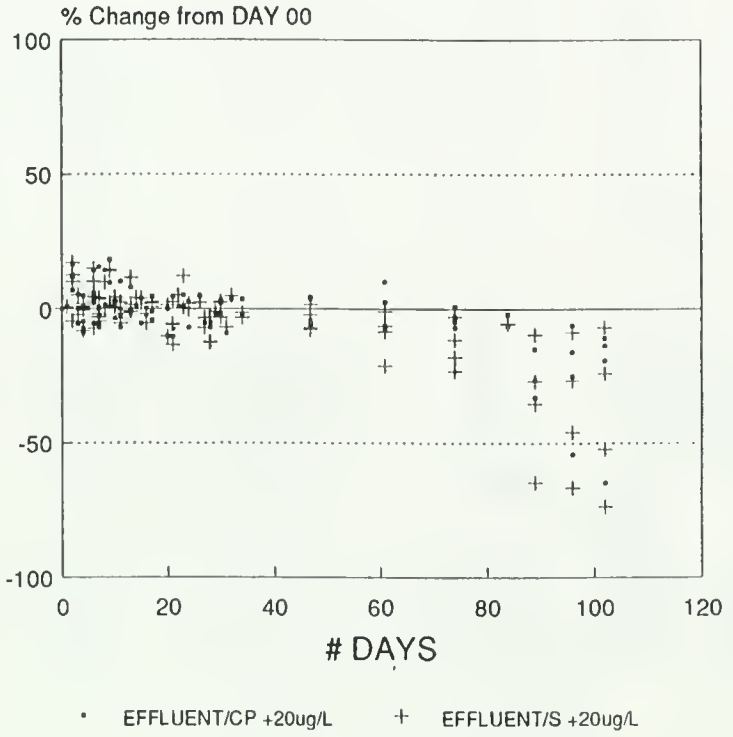


Figure 14B
Extended Time Axis



Stability Study: 4AAP Phenolics